GC-04

Geochemistry
Research
Laboratory
Notebook
VOL. GC-04

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Geochemistry Research Lab
Notebook Vol. GC-04,
Scientific Notebook #020

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Geochemistry Research Lab Notebook

VOL. GC-04

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Pages 1 through 304 of this Scientific Notebook were reviewed for compliance with QAP-001 in response to Corrective Action Request 94-02. Corrections and clarifications were made as appropriate. In some cases, the date of a change will reflect the date of this review rather than the date of the original Scientific Notebook entry.

R Falck QA
SWRE
11/02/94

5/21/91 RTP

Preparation of Zeolite Samples to be Sent to Texas Tech Univ for Chemical Analysis using ICP

Method: Approximately 0.5 g of the zeolite material is taken and placed in plastic vials (except for the CSC sample, of which only 0.15 g is used). The vials are labeled A to J; 2 of the vials have NBS standards. The vial labels and the corresponding sample numbers are listed below:

A – analcime; # ASH*200/300*UC*WA*TTech
B – clinop.; # CSC*HL*Naf*TTech
C – K-feldspar; NBS 70A
~~D~~ E – clinop.; # CDN*100/200*HL*Naf*TTech
~~E~~ F – CDN* 100/200*UC*WA*HL*CPT*TTech
~~F~~ G – CDN*100/200; untreated clinop.
G – heulandite; HPI*TTech
H – Na-feldspar; NBS-99A
I – analcime; ASH*230/325*UC*WA*TTech
J – CDN*100/200*UC*WA*HL*CPT*TTech

5/24/91 T.G.

Preparation of Sodium Form (Naf) Solutions

Objective: Replace all free cations in the Death Valley Junction clinoptilolite with Na^+ ions so isotherm and ion exchange experiments can be performed.

Method: Saturate the CDV in a 3M Sodium Solution @ 90°C

Equipment: (for 480 g CDV)
Versa Bath® S, Model 236 (const. temp. bath)
12 - 500mL Nalgene polypropylene bottles
Large beakers (3 L or 4 L)
Mettler PM 4600, balance accurate to .1g (SN-20467)

Reagents: a.) 3 M Sodium Chloride - dissolve 1051.92 g NaCl in to 6L distilled H_2O. (lot #905522)

$$3 \frac{mol}{L} * 58.44 \frac{g}{mol} * 6L = 1051.92 \text{ g NaCl}$$

Procedure:
1.) Wash 12-500 mL polypropylene (PP) bottles in dilute acid and rinse with distilled H_2O.
2.) Add 400 mL of 3M NaCl to each of the 12-PP bottles. Set the remaining solution aside in a 1L-2L PP bottle for later use.
3.) Weigh 40.0 g CDV and add to a 500 mL PP bottle. Repeat 11 more times.

4.) When all 12 bottles contain 400 mL 3M NaCl solution and 40.0 g CDV place the bottles into a water bath @ 90°C and 50 rpm.
Caution: the water evaporates quickly, so keep filled.

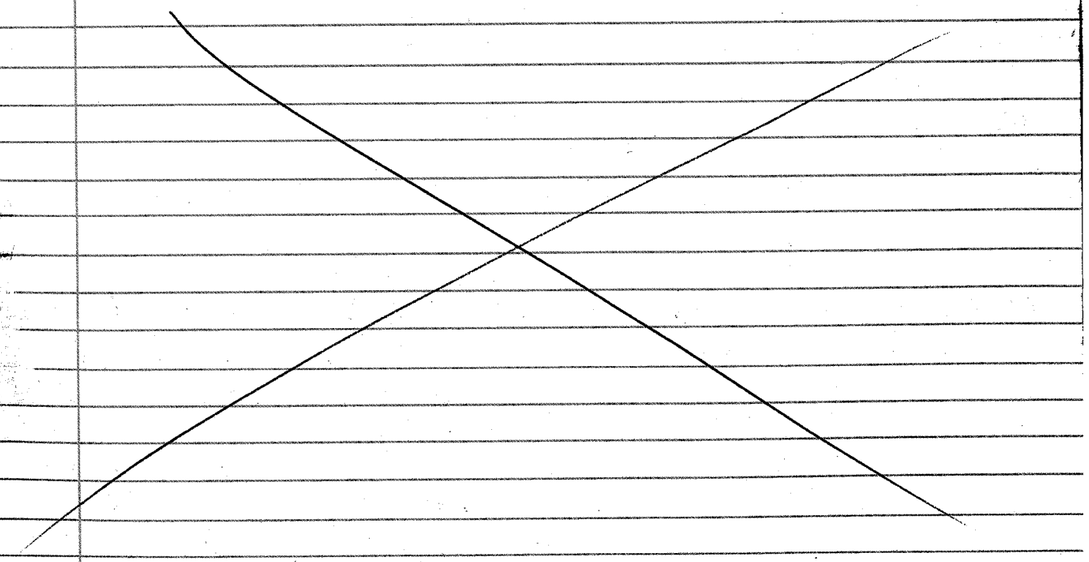
~~5.) Over at least an eleven day period,~~ T.G. 5/24

5.) Replace each bottles solution with a new solution of 3M NaCl by decanting the used solution into a drain. This should happen every other day except on weekends, which allows the clinoptilolite to soak for 3 days instead of 2.

6.) Continue step 5 for @ least eleven days.

6/18 T.G.

7.) Label the bottles (e.g. CDV*100/200*UC*WA*HL*CPT*Naf*A)



5/27/91 RDP

0910 – Sample CDV*100/200*UC*WA*HL*CAT*Na$*CA1 taken from one of the bottles of CDV samples exchanging with 3M NaCl at 90°C. Approximate weight (wet) is 4.4 g. The sample was rinsed 5X with DeI water; test for Cl⁻ using AgNO3 was negative after the 5th rinse. Sample was placed in 60 ml polypropylene bottle, capped & placed in shaker bath at 90°C.

1600 – Take bottle containing CDV*...*CA1 out of water bath. ~~Rinse~~ RDP 5/28/91 Test solution for Cl⁻; positive result. Rinse. Next Cl⁻ test negative. Replace water & put back in bath.

5/28/91 RDP

0945 – Take out CDV*...*CA1 from bath. Test for Cl⁻ is negative. Rinse. Decant powder into 20 ml beaker. Dry in Oven at ~90°C.

1620 – Take beaker of CDV*...*CA1 out of oven; cover w/ parafilm. Let cool to room temp. Weight = 16.6715 g (container + film + powder). Equilib with water vapor over sat'd NaCl in desiccator.

5/29/91 RDP

1100 – Sample CDV*100/200*UC*WA*HL*CAT*Na$*CA2 was taken from a bottle with CDV exchanging with 3M NaCl. Approx. weight of 4.0 g (wet sample). Rinsed w/ DeI H2O 5X – Cl⁻ test w/ AgNO3 is positive. Rinsed again, giving negative Cl⁻ test. Replace H2O (in 60ml poly propylene bottle); then keep in shaker water bath at ~90°C.

1330 – Weight of CDV*100/200*UC*WA*HL*CAT*Na$*CA1 = 16.5634. Lost ~ 0.1 g. since yesterday.

1450 – CDV*...*CA2 taken out of bath. Water decanted and checked for Cl⁻. Positive result. Rinse 2X. Negative Cl⁻ test. Replace DeI H2O and put back in water bath at ~90°C.

5/27/91 0800 T.6.

Refer to pg 3 -
Each polypropylene bottle was decanted. A new solution of 3 M NaCl (lot # 905522) was made. 400 ml of this solution was placed into each of the 12 ~~test tubes~~ bottles. Each bottle was placed back into the 90°C water bath for another 2 days. Ref. 11 water bath.
T.6 5/27
NOTE: Don't decant off any clinoptilolite.

5/29/91 0800 T.6

Refer to pg 3 -
The polypropylene bottles old (3M NaCl) solutions were decanted and replaced with new 3M NaCl (lot # 905522) solutions. 400 ml of the new Sodium Chloride solution were placed into each of the bottles and set back into the 90°C water bath for another 2 days.
T.6 5/29 This was also performed on the 1st & 2nd Na's
Also, just because the ~~machine~~ water path T.6 5/29 is set @ 50 rpm doesn't necessarily mean that the clinoptilolite gets swirled enough, so after each bottles solution is replaced, shake up the CDV sediment in order to 'rotate' the top layer and then place the bottle back into the water bath.
Refill water bath.

5/30/91 D.M.
Preparation of 0.5, 0.05 0.005, and 0.0005 M solutions of NaCl, KCl + $CaCl_2 \cdot 2H_2O$ to be used for testing stability + reproducibility of electrodes in ion-selective analysis Go to page 12

5/30/91 0800 T. 6.

Fill water bath that is holding the 40g CDV.

5/30/91 0815 T. 6.

Na^+ electrode practice

Purpose - to become aquainted with the Expandable Ion Analyzer (EA920), Sodium electrode, and its respective reference electrode.

Method - Direct calibration using 2 standards to bracket the range. This method measures a large number of samples easiest.

Equipment - Expandable Ion Analyzer EA920 S/N S00IA
Ross Sodium Electrode Orion Cat No. 209574-A02
Ross Ref. Electrode Orion Cat No. 800500
Corning Stirrer
Magnets (various sizes)
Pipets (" ")

Reagents - a.) 1000 ppm Sodium Standard Orion Cat No 841100
b) Sodium 'ISA' Orion Cat. No 841111
(Ionic Strength Adjustor)

c.) .5 M NaCl - Dissolve Sodium Chloride (lot # 98552) 7.305 g NaCl, in 250 ml distilled H_2O.

$$.5\frac{mol}{L} * 58.44\frac{g}{mol} * \frac{1L}{1000mL} * 250mL = 7.305\ g\ NaCl$$

Procedure - 1) Prepare standards with composition similar to that of the ~~sample~~ TG 7-24-94 to be analyzed. Add 10 ml ~~for ever~~ ISA for every 100 ml Sample e.g. For samples whose average concentration is about 300 ppm, prepare two standards. The first with 50 ppm and the second with 500 ppm. NOTE the difference of X10 between the two standards.

2.) From the .5 M NaCl solution, make up various molar concentrations ranging from .0005 → .5 M and analyze them. The meter should be recalibrated when solution reading (ppm) lies outside the currently calibrated rauge.

3.) For each measurement wash the electrodes with ~~the~~ TG 7-24-94 Sodium wash before placing them into a new solution. Read the meter in ppm's.

Conversions:

1 M	≅ 23,000	ppm
.1 M	≅ 2300	ppm
.05 M	≅ 1150	ppm
.005 M	≅ 115	ppm
.0005 M	≅ 11.5	ppm

etc.

TG 7-24-94

4) ~~Add~~ To analyze solutions with a molar concentration much lower than .5 M NaCl perform a series of dilution steps. Take ~~exactly~~ TG 7-24-94 10 ml of the .5 M NaCl and dilute to 100 ml with distilled H_2O.

5.) To analyze solutions with a molar concentration much lower than .05 M NaCl, take 10 ml of .05 M solution and dilute to 100 ml with 90 ml H_2O. This produces a .005 M NaCl solution.

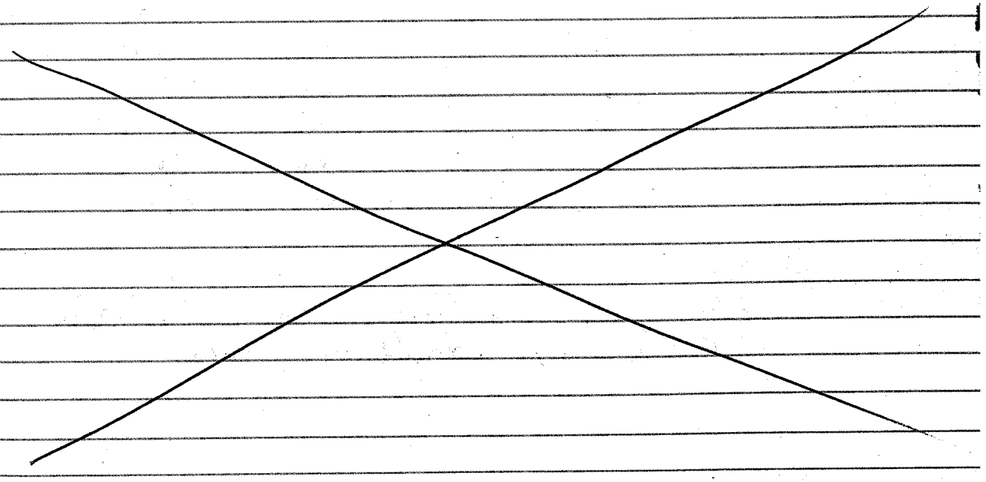
6.) Repeat ~~of~~ TG 7-24-94 5.) ~~if~~ TG 7-24-94 if a lower concentration is wanted, ~~H~~ except use the .005 M solution instead of the .05 M one.

General Eq:

$$\text{~~.05~~ concentration (M)} * \frac{\text{vol (concentration)}}{\text{vol (conc.)} + \text{vol } (H_2O)} * X(\text{ppm}) = y\ (\text{ppm})$$

e.g.

$$.05\ M\ NaCl * \frac{.2\ ml\ NaCl}{\underbrace{2\ ml\ NaCl + 9.8\ ml\ H_2O}_{\text{total of 10. can have any total}}} * 23000\ \text{ppm Na} = 23\ \text{ppm Na}$$



5/31/91 T.G.

Results Na+ Results. (1st Run)

diluted to 10 ml (total)

ml of 0.005M NaCl	theo (ppm)	exp (ppm)	error (%)
1	11.5 ppm	11.4	.8%
1.5	17.25 ppm	17.1	.9%
2	23 "	22.2	3.5%
5	57.5 "	55.9	2.8%
7	80.5 "	80.4	.1%
10	115 "	113	2%

ml of 0.05M NaCl	theo (ppm)	exp. (ppm)	error (%)
1	115 ppm	113	2%
1.5	172.5 ppm	175	1.5%
2	230 ppm	240	4%
5	575 ppm	579	.7%
8	920 ppm	915	.7%
10	1150 "	1140	.8%

ml of 0.5M NaCl	theo. (ppm)	exp. (ppm)	error (%)
1	1150	1140	.8%
1.5	1725	1670	3%
2	2300	2250	2%
5	5750	5690	1%
8	9200	8830	4%
10	11500	11100	3%

The preceeding concentrations were made by serial dilutions

5/31/91 T.G.

Na+ Results (2nd Run) (approx. 1 day later)

diluted to 10 ml (total)

ml of 0.005M NaCl	theo (ppm)	exp. (ppm)	error (%)
1	11.5	11.3	1.7%
1.5	17.25	16.7	3.2%
2	23	22.1	4%
5	57.5	56.4	2%
7	80.5	77.4	4%
10	115	111	3%

ml of 0.05M NaCl	theo (ppm)	exp. (ppm)	error (%)
1	115	111	3%
~~2~~ 1.5	172.5	174	.9%
2	230	240	4%
5	575	580	.9%
8	920 ~~805~~	924	.4%
10	1150	1160	.8%

ml. of 0.5M NaCl	theo. (ppm)	exp. (ppm)	error (%)
1	1150	1160	.8
1.5	1725	1760	2%
2	~~1760~~ 2300	2360	2.6%
5	~~2360~~ 5750	~~8860~~ 5860	2
8	~~5860~~ ~~8050~~ 9200	8860	3.7
10	~~8860~~ 11500	11900	3.5

move one line down

TG 7-24-94

wrong column

T.G. 5/31

5/30/91 P.M (0.5) Refer to page 7

A ~~5.0~~ M solution of NaCl was prepared by adding 7.304(9)g NaCl to 250ml of water. 0.05M, 0.005M, and 0.0005M solutions were prepared from this by serial dilution. NaCl stock # is 905522.

A 0.5M solution of KCl was prepared by adding 9.319(3)g % of KCl to 250 ml of water. 0.05M, 0.005M and 0.0005M KCl solutions were prepared from this by serial dilution. KCl stock # is 885967.

A 0.5M solution of $CaCl_2 \cdot 2H_2O$ was prepared by adding 18.376(5)g of $CaCl_2 \cdot 2H_2O$ to 250ml of water. 0.05M, 0.005M and 0.0005M solutions were prepared from this by serial dilution. $CaCl_2 \cdot 2H_2O$ stock # is 4160 KENN. All solutions were stored in acid-washed plastic bottles.

5/30/91 RJP

1000 – Sample CDV*100/200*UC*WA*HL*CPT*NaS*CA2 removed from water bath. Solution tested for Cl^-. Slight positive result. Rinsed 2x and decanted. Then dried in oven at ~50°C.

1500 – Remove CDV*...*CA2 from oven. Let cool. Weight = 16.3539 g. Store in dessic over sat'd NaCl. ~~Weigh~~

Weigh Sample CDV*...*CA1. Wt = 16.5464; showed further decrease from original

5/31/91 RJP

0845 – Sample CDV*100/200*UC*WA*HL*CPT*NaS*CA3 taken from one bottle exchanging with 3M NaCl at ~90°C. Approx. weight (wet) = 4.5 g. Rinse 5x with deI H_2O. Test for Cl^-: slightly positive. Rinse 2x. Replace deI H_2O.

1030 – Put sample CDV*...*CA3 in shaker bath at 90°C.

6/3/91 RJP

0900 – Sample CDV*100/200*UC*WA*HL*CPT*NaS*CA3 taken out of water bath. Cl^- check negative. Rinse and decant into 20 ml beaker and dry in oven at ~50°C.

0915 – Sample CDV*100/200*UC*WA*HL*CPT*NaS*CA4 taken from one bottle of CDV*...*NaS reacting w/ 3M NaCl. Approx weight (wet) = 5.58g. Rinse 5x w/ deI H_2O. Cl^- test positive. Rinse 2x. Replace H_2O and put in water bath at ~90°C.

1600 – Sample CDV*...*CA4 taken out of bath. Cl^- test positive. Rinsed 2x. Replace H_2O. Put back in bath at ~90°C.

6/4/91 RJP

1025 Take sample CDV*...*CA3 out of oven. Let cool. Weight = 16.430 g (including beaker + parafilm). Place open in dessicator over sat'd NaCl soln.

Reweigh CDV*...*CA1 = 16.5447 g.

Reweigh CDV*...CA2 = 16.5426 g.

1040 Sample CDV*...*CA4 taken out of water bath. Cl⁻ test negative. Rinse 2X. Decant powder into 20 ml beaker. Dry in oven at ~50°C.

6/3/91 T.G. ~~8:00~~ 0800
Fill water bath containing CDV.

6/3/91 T.G. ~~8:15~~ 0900

Changed 3M NaCl solution in the 500 ml bottles with a new solution of 3M NaCl.
The old 3M NaCl solutions in the poly-propylene bottles were decanted and replaced with new 3M NaCl (lot # 7581 KEME) solutions. ~~400~~ 400 ml of the new Sodium chloride solution were placed into each of the bottles and set back into the 90°C water bath. They were ~~first~~ shaken before placed

back in.
5/31/91 On this day, the 3M solution was changed. I forgot to write it in.
T.G.

6/5/91 RJP

1000 - Sample CDV*100/200*UC*WA*HC*CPT*Naf*5 taken from one bottle of CDV*...*Naf exchanging with 3M NaCl at 90°C. Approx. weight (wet) ~ 4.9 g. Rinse 5X. Cl⁻ test

Take sample CDV*...*CA4 out of oven. Cover w/ parafilm. Let cool.
RJP 6/5/91 ~~Weight = 17.2080 g. Store in dessicator over sat'd NaCl soln.~~
Label; ~~Weight~~ = 17.3234 g.
Store in dessicator over sat'd NaCl soln.

Reweigh: CDV*...*CA1 = 16.5449
CDV*...*CA2 = 16.5443
CDV*...*CA3 = 16.5081

6/5/91 0800 T.G.
Decanted solutions (3M NaCl) from the 12-500 ml polypropylene bottles. Refilled with TG 7-24-94 a each bottle with TG 7-24-94 pure distilled (H_2O and placed them back in to the waters bath.

6/5/91 1000 T.G.
Took all twelve bottles back out of the waters bath and washed TG 7-24-94 each all the chloptiblete within each of the 500 ml bottles.
Decanted each bottle and replace with a new 400 ml distilled Waters. Shake to mix the CDV and allow to settle. Repeated the above step 6 times for each bottle.

After the CDV in each of the 12 bottles has been washed six times, ~~place them~~ TG 7-24-94 T.G 6/5 check each one for chloride. Decanted ≈5-10 ml into small beaker and added (lot # CO74) .1 N $AgNO_3$. Watch for any white precipitate (ppt.). If a white ppt. formed, wash the CDV in each bottle 2 more times and then check for chloride again. When no more of the solutions form a Silver chloride ppt any more, place the 500 ml bottles back into the 90°C waters bath. ~~until the next morning.~~ T.G 6/5

6/5/91 1500 T.G.

After allowing the CDV to sit in the 90°C water bath, recheck for chloride by decanting ≈5-10 ml into a small beacker add a couple drops of the .1 N $AgNO_3$ (lot # CO74) If any precipitate TG 7-24-94 ($AgCl$) forms, wash that particular ~~500 ml~~ 40g CDV in the 500 ml bottle, with new distilled water, at least twice.
Do all twelve bottles until no white precipitate occurs in any.
Set the bottles back in to the 90°C waters bath and allow to set overnight.

6/6/91 0800 T.G.

Take out each the 12 polypropylene bottles from the waters bath and check for chloride by decanting approximately 5-10 ml into a small beaker. Add a few drops of .1 N $AgNO_3$ (lot # CO74) to the beaker TG 7-24-94 and watch for the slightest change. ~~in color~~ If a white cloud appears where the drops are added or if the water turns cloudy (at all), change the solution in the bottles and place back into the waters bath.

6/6/91
1600 – MJP

Take out the 12 bottles of CDV*...*Na from the water bath. Swirl each bottle and ultrasonically clean for about 5 minutes. Decant the solution and replace with deI H_2O. Repeat ultrasonic cleaning. Do 4X. Then give a final rinse with deI H_2O and transfer into ~~250~~ MJP 6/6/91 20–250 ml beakers the contents of the 12 bottles. Let settle, decant, and dry in the oven overnight at about 65°C.

Note: (1) First 4 rinsings were done with deI H_2O w/ resistivity between 8 & 9 megaohm. The filters in the lab Nanopure unit need replacement. The final rinse was done w/ deI H_2O taken from the MilliQ system in Div. 01.

(2) The decanted solutions throughout the rinsing procedure were cloudy, possibly due to disaggregation of the clinoptilolite grains into finer grain sizes.

(3) Chloride tests of each bottle after taking them out of the water bath are all negative.

1630 – Take sample CDV*200/200*UC*WA*HL*CDT out of water bath. (*Na*CA5
Cl^- check is negative. Rinse 2X. Transfer into 20 ml beaker. Dry in oven at ~65°C.

6/7/91
0800 T.G.

Practice with Ion Selective Electrodes:
Na^+, K^+, Ca^{2+}

Objective – to check reproducibility.

Method – Direct calibration.

Equipment – Expandable Ion Analyzer EA920: S/N S001A & S/N TV64A
Ross Sodium Electrode Orion Cat. # 09574-A02
Ross Reference Electrode Orion Cat. No. 800500
Potassium Elec. w/BNC Orion Cat. # 9319 BN
Ref. Electrode DBL Junct. Orion Cat. No. 900200
Replacement K^+ Sensing Module " 931901
Calcium Elec. w/BNC " 9320BN
Ref. Electrode Single ~~Elec.~~ Junct. " 900100
Replacement Ca^{2+} Sensing Module " 932001
Corning Stirrer
Magnets (various sizes)
Pipets (" ")

Reagents – a.) 1000 ppm Sodium Standard Orion Cat. No. 841108
100 ppm " " " 941107
Sodium 'ISA' " 8411 "

See pg 12 { .5M NaCl, .05 M NaCl, .005 M NaCl
.0005 M NaCl

b.) .1M Potassium Standard CORNING Cat No. 478164
Potassium ISA Orion cat No. 931911
see pg 12 { .5M KCl, .05M KCl, .005M KCl
.0005M KCl

a.) .1M Calcium Chloride Standard Orion Cat No. 922006
Calcium ISA " " 932011
see pg 12 { .5M $CaCl_2$, .05M $CaCl_2$, .005M $CaCl_2$
.0005M $CaCl_2$

Procedure - 1.) ~~Prepare~~ TB 7-24-94 Calibrate the meter, Ion Analyzer by preparing two standards that bracket the known concentration by one decade only. ~~After~~ TB 7-24-94 That is, if

2.) when each concentration has been recorded by a one decade bracket, then recalibrate the meter to go over all 3 decades and read the meter allow the meter to extrapolate to the .5M concentration and record observations.

3.) Follow the instructions from each of the owners manuels and pay attentions to the hints, i.e. ~~use~~ always use ISA in all samples and standards.

Since the ion selective electrodes and their respective reference electrodes can fit in 20 ml plastic ware, your samples and standards should only be about 10-15 ml. Using any more standard or sample is wasteful.

Results Na^+

	(M) conc $(NaCl)_{KCl}$	(ppm) Theo	(ppm) exp (5/31)	(5/31) error (%)	(6/4) exp.	6/4 error
recalibrated for each decade (x/o)	.5 M	11500			12100	5
	.05 M	1150	1170	1.7 %	1160	.9
	.005 M	115	123	7 %	124	8
	.0005 M	11.5	13.3	16%	13.5	17
cal. titrated over entire range	.5	11500	TB 7-24-94 11700	TB 7-24-94 1.7	13000	13
	.05	1150	TB 7-24-94 1080	TB 7-24-94 6	1150	0
	.005	115	TB 7-24-94 115	TB 7-24-94 0	114	.8
	.0005	TB 7-24-94 11.5	TB 7-24-94 13.3	TB 7-24-94 16	13.3	16
	.0001					

.0005: Since the .0005M NaCl virtually showed the same results each time, I was led to believe that 13.3 ppm was indeed the concentration.

.005: At this concentration, TB 7-24-94 ~~I would~~ the meter interpreted ~~the~~ it ~~over~~ best over the entire range. And the .005 concentration ~~wasn't~~ didn't lie on the linear line between the one decade calibration.

.05: All of the results were exceptional except one which may have been the operators fault

.5: error exists here because the meter extrapolated to reach the concentration.

Results K^+

	Conc. (M)	Theo. (ppm)	6/4/91 exp (ppm)	6/4/91 error (%)	6/6/91 exp (ppm)	6/6/91 error (%)
calibrated over each decade (x10)	(KCl)					
	.5	19550	18000	8	17600	10
	.05	1955.	1950	.26	1930	1.2
	.005	195.5	190	2.8	187	4.3
	.0005	19.55	18.6	4.9	18.4	5.8
calibrated over entire range	.5	19550	18900	3.3	18800	3.8
	.05	1955	1900	2.8	1880	5.8
	.005	195.5	175	10	173	11.5
	.0005	19.55	15.8	19	15.5	20.1
	.001					

.5 - this high concentration ~~provided~~ caused a large error. It was not diluted because I was already measuring a serial dilution. The best results @ this concentration were ~~read~~ found when the solution was calibrated over the active range of solutions. On the other hand, calibration over the entire range also provide the worst results @ low concentrations.

All results were ± 2% reproducible providing they were done right (i.e, a K^+ replacement sensing module was used)

Results Ca^{2+}

	Conc. (M) $CaCl_2$	Theo (ppm)	6/5/91 exp. (ppm)	6/5/91 error (%)	6/6/91 exp (ppm)	6/6/91 error (%)
calibrated over each decade (x10)	.5	20050	18400	8.2	18700	6.7
	.05	2005	1980	1.2	1960	2.2
	.005	200.5	197	1.7	198	1.2
	.0005	20.05	19.1	4.7	18.5	7.7
calibrated over entire range	.5	20050	18600	7.2	19000	5.2
	.05	2005	2060	2.7	2040	1.7
	.005	200.5	211	5.2	208	3.7
	.0005	20.05	18.8	6.2	18.4	8.2

The same type of error was seen here as in the K^+ results. At high concentrations, > .1 M the meter extrapolates better over the larger range, yet at the bottom of the range, more error is seen.

All results, except one, were within the ± 2% reproducibility range. The exception ~~was in the low~~ occured @ .0005 M conc. And although the the numbers "are close", the number is small and ~~prod~~ created the 3% error.

6/7/91 RDP

0900 — CDV*..*Nafsamples in oven were taken out. The beakers were covered with parafilm, and labeled CDV*...*NafA through CDV*...*NafJ (Sharpie pen for CDV* 100/200 UCLEWAR HLE PT*NafA). The samples were let to cool, then weighed.

Sample	Wt. Sample (+beaker+cover)
CDV*..*NafA	166.0075 g
" *B	168.9270 g
" *C	143.9728 g
" *D	153.6124 g
" *E	158.7105 g
" *F	147.8252 g
" *G	154.8566 g
" *H	133.1551 g
" *I	134.7728 g
" *J	140.0821 g

1030 — Beakers were left open, then samples were equilibrated with water vapor over saturated NaCl in desiccators (5 beakers per desic). Desiccators were placed on top of gyratory shaker to accelerate equilibrium water absorption.

1050 — Take sample CDV*..*Naf*CA5 out of oven. Cover beaker with parafilm, let cool. Weight = 16.8580 g. Store in desiccator over sat'd NaCl solution.

Re-weigh:

CDV*..*Cal	= 16.5449 g
...*Ca2	= 16.5442 g
...*Ca3	16.5093 g
...*Ca4	17.4197 g

T.O.	Time (1500) T.O. 6/7/91	(0830) T.O. 6/10	(1645) T.O. 6/10	(0800) T.O. 6/11	(1600) RDP 6/11
1500					
CDV*..*A	166.2057	167.7678	167.8531	168.0697	168.1061
CDV*..*B	169.1356	170.8095	170.8591	171.0078	171.0269
CDV*..*C	144.1679	145.4414	145.4580	145.5057	145.5166
CDV*...*D	153.8010	155.2680	155.3246	155.4650	155.4835
CDV*...*E	158.9172	160.4043	160.5033	~~149.1368~~ 160.6962 T.O. 6/11	160.7244
CDV*...*F	148.0283	149.3363	149.3630	149.4368	149.4408
CDV*...*G	155.0567	156.5773	156.6357	156.7857	156.8030
CDV*...*H	133.3415	134.2573 T.O. 6/10	134.2535	134.2960	134.2882
CDV*..*I	134.9681	135.9520	135.9653	136.0092	136.0233
CDV*...*J	140.3087	141.5034	141.5131	141.5567	141.5581

Beakers are taken out of desiccators and weighed. To weigh, take the beakers and ~~cover~~ completely cover the top with the other half of the parafilm. Once weighed, flip the parafilm halfway back and place back into the dessicators after recording the weight. Repeat A-J. To 6-24-91 Ate To 7-24-91

Repeat this everyday until successive weights occur for every sample (beaker + sample + parafilm).

6/10/91 TDD

0930 — Reweigh samples CDV*...*CA1 to 5

Sample #	Total Weight
CDV*100/200*UC*WA*HL*CPT*NaS*CA1	16.5453
CDV*. *CA2	16.5453
CDV* *CA3	16.5099
CDV* *CA4	17.4211
CDV* *CA5	16.9801

0945 — Take some of the CDV*...*CA5 sample for SEM analysis, to check potential presence of secondary phases. Reweigh = 16.9670

6/12/91 0800 T.6

TIME — DAY	0800 6/12/91	0800 6/13/91	0800 6/14/91	0800 6/17/91	0800 6/18/91	0930 6/19/91	0800 6/21/91
CDV*...*A	168.2216	168.3438	168.3739	168.4033	168.4090	168.4049	168.4002
CDV*...*B	171.1387	171.1699	171.2076	171.2387	171.2405	171.2383	171.2152
CDV*...*C	145.5461	145.5822	145.5912	145.6189	145.6142	145.5970	145.5923
CDV*...*D	155.5625	155.6086	155.6084	155.6458	155.6321	155.6240	155.6077
CDV*...*E	160.8391	160.8804	160.8959	160.9729	160.9537	160.9492	160.9423
CDV*...*F	149.4735	149.5024	149.5124	149.5124	149.5164	149.5116	149.4945
CDV*...*G	156.8796	156.9364	156.9452	156.9796	156.9726	156.9646	156.9445
CDV*...*H	134.3081	134.3158	134.3072	134.3153	134.3103	134.3024	134.2945
CDV*...*I	136.0473	136.0804	136.0597	136.0716	136.0710	136.0628	136.0577
CDV*...*J	141.5956	141.6032	141.6037	141.6106	141.6182	141.6074	141.5944

Took samples out of dessicator and weighed @ least once a day until the sample wts (sample + paraffin + beaker) remained the same. See pg 34. T.6 6/21/91

6/14/91 T.6 1100

Preparation of 0.5N NaCl-KCl & 0.5N NaCl-$CaCl_2$ Solutions

Objective — To prepare various NaCl-KCl & NaCl-$CaCl_2$ solutions, but with each solution having a total normality of 0.5N.

Method — weigh the compounds using the previously calculated weights which were predicted before the experiment. An example can be found at the end ~~of this write up~~. T.6 6/14 pg 29.

Equipment — Mettler AE 240, balance accurate to .001g (101237)
weighing bottles (Pyrex 20 & 100ml w/lids)
Metal spatulas
beakers (50 → 600ml)
500 ml volumetric flasks
eye dropper
Corning stirrer
magnets
magnetic rod
20 - 500 ml Nalgene polypropylene bottles.

Reagents — Potassium Chloride lot # 885967
Sodium Chloride lot # 905256A
Calcium Chloride Dihydrate lot # 4160 KENN
Distilled Water @ 17.3 MΩ-cm
T.6 6/14 (deionized)

Procedure — (1) Prepare 500 ml of NaCl-KCl (NaCl-$CaCl_2$) aqueous mixture with a total normality of 0.5N and a fixed Na/K (or Na/Ca) ratio by taring reagent grade KCl and $CaCl_2 \cdot 2H_2O$

in the amount given in Table 1 (or 2) onto a clean (acid-washed) 600 ml beaker. Add about 200 ml deionized water to the beaker prior to adding the reagents, that way, the weighed reagents which absorb moisture from the air (e.g. $CaCl_2 \cdot 2H_2O$) can be added directly to the water. Note - Use weighing bottles when weighing the calcium chloride to prevent water absorption since this will increase the $CaCl_2$'s weight as you are weighing it.

2.) Once ~~the~~ 6/19 T.6 one of the KCl-NaCl ($CaCl_2$-NaCl) is weighed and placed into solution, add a stir bar and dissolve the salts completely using a stirrer (Add more water if necessary)

3.) Retrieve the stir bar with a magnetic rod, then carefully rinse the rod and stir bar with deionized waters. Decant the solution into a clean 500 ml volumetric flask (with help of a glass rod); rinse the beaker (and glass rod) with deionized water several times, carefully transferring all washings into ~~the~~ 6/4 T.6 the volumetric flask.

4.) Then fill the flask to about 2-3 inches below the mark with deionized water; swirl or shake the flask, let stand for a few seconds, then add water dropwise up to the mark; remix.

5.) Transfer the solution into a clean, acid-washed and dry ~~volumetric~~ 6/14 T.6 500 ml polypropylene bottle. Label the bottle (e.g. NaCl/KCl*0.5N* $0.1X_K$ plus date and initials)

6.) Then take approximately 5 ml from each bottle and measure the pH.

	X K(.5Nsoln)	Wt. KCl/500ml	actual wt KCl	Wt. NaCl/500ml	actual wt NaCl
1	0.1	1.8638	1.8636	13.15	13.1503
2	0.2	3.7276	3.7277	11.689	11.6892
3	0.3	5.5913	5.5912	10.228	10.2282
4	0.4	7.4551	7.4551	8.7664	8.7664
5	0.5	9.3189	9.3190 6/14	7.3054	7.3055
6	0.6	11.183	11.1832 T.6	5.8443	5.8445
7	0.7	13.046	13.~~1832~~ 0462	4.3832	4.3833
8	0.8	14.91	14.9100	2.9221	2.9222
9	0.9	16.774	16.7742	1.4611	1.4612
10	1	18.638	18.6382	0	—

Table 1
0.5 N KCl - NaCl
500 ml soln

e.g.

$$M_K = \frac{1.8638\text{g KCl} / 74.55\text{g KCl/mol KCl}}{500\text{ml}} * \frac{1000\text{ml}}{1\text{L}} = .0500 = M_K$$

$M_K = .5 * X_K \Rightarrow X_K = \frac{.05}{.5} = .1 \Rightarrow X_K = .1$

$$M_{Na} = \frac{13.15\text{g NaCl} / 58.44\text{g NaCl/mol NaCl}}{500\text{ml}} * \frac{1000\text{ml}}{1\text{L}} = .4500$$

$M_{Na} = .5 * X_{Na} \Rightarrow X_{Na} = \frac{.4500}{.5} = .9000$

Note: $M_K + M_{Na} = .5 \Rightarrow .05 + .45 = .5$ TN

$X_K + X_{Na} = 1 \Rightarrow .1 + .9 = 1.0$

	X_Ca(.5Nsoln)	WtCaCl2DH	actual $wt CaCl_2 \cdot 2H_2O$	WtNaCl	actual wt NaCl
1	0.1	1.8377	1.8379	13.15	13.1498
2	0.2	3.6754	3.6766	11.689	11.6892
3	0.3	5.5131	5.5129	10.228	10.2283
4	0.4	7.3508	7.3507	8.7664	8.7665
5	0.5	9.1885	9.1888	7.3054	7.3055
6	0.6	11.026	11.0262	5.8443	5.8444
7	0.7	12.864	12.8636	4.3832	4.3833
8	0.8	14.702	14.7025	2.9221	2.9221
9	0.9	16.539	16.5393	1.4611	1.4611
10	1	18.377	18.3773	0	—

Also; $2 * M_{Ca} + 1 M_{Na} = 0.5N$

Table 2

0.5N $CaCl_2$ – NaCl

500 ml soln.

6/14/91 T.G

1400

X_K (.5N soln)	pH (≈22°C)	temp(°C)	X_{Ca} (.5N)	pH (≈22°C)	temp(°C)
	5.96				
0.1	6.03		0.1	5.88	
0.2	6.04		0.2	5.89	
0.3	5.93		0.3	5.87	
0.4	5.97		0.4	5.94	
0.5	6.00		0.5	5.94	
0.6	5.99		0.6	5.84	
0.7	5.94		0.7	5.89	
0.8	5.99		0.8	5.86	
0.9	6.03		0.9	5.88	
1.0			1.0	5.91	

6/18/91 0830 T.G.

Preparation of 0.05N NaCl-KCl & 0.05N NaCl-$CaCl_2$ Solutions

Objective - To prepare various NaCl-KCl & NaCl-$CaCl_2$ solutions, but with each solution having a total normality of 0.05 N.

Method - serial dilution from 0.5 N solutions.

Equipment: 20 - 1 L - polypropylene NALGENE bottles
100 ml pipet and bulb.
~~deionized H_2O~~ T.G 6/18
1000 ml volumetric flask(s)
eye dropper

Reagents - previously made 0.5N NaCl-KCl (NaCl-$CaCl_2$) solutions
> 25 L deionized H_2O

Procedure - 1.) Make up 1000 ml of 0.05N NaCl-KCl (or NaCl-$CaCl_2$) solution by pipetting 100 ml of the 0.5N solution (see pg 27) and diluting it with deionized water in a 1000 ml volumetric flask.
T.G. ~~2.)~~ NOTE: Make sure to rinse the pipet after each use, with deionized H_2O. Dry it (partially) and rinse it with ≈ 10 - 20 ml of solution by pipetting the 0.5N solution ~~(2x)~~ T.G into ~~the~~ T.G. 6/18 the pipet each time. Also, if limited to a few volumetric flasks, rinse after each solution by swirling @ least 3-4

T.6 6/18/91
times with de-ionized water to limit carryover of previous solutions. These do not need to be dried.

2.) Transfer the 0.05 N solution into a 1000 ml polypropylene bottle. Label the bottle (e.g. NaCl/KCl * 0.05 * 0.1 X_K) plus date and initial

3.) Take ≈5ml and measure the pH.
4.) Cover the bottle w/ Kimwipe papertowels and agitate in shaker bath over several days. Remeasure the pH in a couple days.

6/18/91 T.6 0845

≈Temp @ 25.3°C	X_K (.05N solution)	pH	X_{Ca} (.05N soln)	pH
	0.0 T.6	6/19 T.6 ~~5.76~~ 6.08	0.1	5.76
	0.2	6.30	0.2	5.70
	0.3	6.14	0.3	5.68
	0.4	6.03	0.4	5.67
	0.5	6.09	0.5	5.67
	0.6	6.09	0.6	5.67
	0.7	6.17	0.7	5.68
	0.8	6.06	0.8	5.76
	0.9	6.14	0.9	5.73
	1.0	6.00	1.0	5.68

the pH was actually measured of 6/17/91 @ 1600.

6/18/91 T.6. 1330
Remeasured the pH of the 0.5N NaCl-KCl ($NaCl-CaCl_2$) solutions again for the 1st calculations, see page 30.
Avg. temp was 23.7°C

T.6. X_K (.5N)	pH	X_{Ca} (.5N)	pH
.1	6.29	.1	5.92
.2	6.22	.2	5.94
.3	6.23	.3	5.91
.4	6.23	.4	5.92
.5	6.18	.5	5.93
.6	6.19	.6	5.92
.7	6.31	.7	5.93
.8	6.19	.8	5.91
.9	6.23	.9	5.89
1.0	6.26	1.0	5.92

6/19/91 T.6 1530
Remeasured the pH of the .05N solutions. See pg 32.

X_K (.05N)	pH
.1	5.62
.2	5.58
.3	5.62
.4	5.56
.5	5.54
.6	5.62
.7	5.57
.8	5.62
.9	5.58
1.0	5.63

~~6/20/91 11~~ X_{Ca} (.05N) pH
was done next day

6/20/91 1400 T.G.
Remeasured the pH of the 0.05 N solutions
See pg 32
avg. temp ≈ 24.5°C

X_{Ca} (.05 N)	pH
.1	5.61
.2	5.57
.3	5.54
.4	5.56
.5	5.52
.6	5.59
.7	5.57
.8	5.59
.9	5.59
1.0	5.58

6/21/91 0800 T.G.
After June 18, ~~the~~ T.G. the dessicators were taken off the gyrotory shaker-model G2, which were used to increase the equilibriation rate. They, the beakers inside the dessicators, were taken off because they were believed to be in equilibrium. Note though that over the next 3 days, the beakers labeled CDV*...*A-I all lost weight.

6/21/91 0845 T.G.

Each beaker, label CDV*...*A-J, was added to a jar and mixed in order to create a homogeneous mixture of clinoptilolite. The jar was labeled,
CDV*...*Naf*A-J (mixed)
and the weight of the CDV was 459.87 g after equilibration in saturated NaCl

At that time, Jim Prikryl took approximately 0.05 g for SEM Analysis
(Scanning Electron Microscope)

6/21/91 1350 JP
Sample CDV*100/200*UC*WA*HL*CPT*Naf*A-J renamed CDV*100/200*UC*WA*HL*CPT*Naf and entered into Sample custody log.

6/21/91 1355 JP
Three ~5 g samples of CDV*...*Naf were taken for surface area analysis by Micromeritics Material Analysis Laboratory. (See GC-03/203 for labelling).

6/24/91 T.G. 0800

Preparation of the CDV--NaCl/KCl (T.G. 0.5N) Mixtures for Isotherm Experiment @ 25°C

Objective - to prepare 0.5N KCl-NaCl-CDV ~~solutions~~ mixtures -- where CDV is CDV*100/200*UC*WA*HL*CPT*Na and .5N KCl-NaCl is on pg 27 Vol 4 -- by adding various amounts of zeolite to specific solution volumes. T.G. 7-24-94

Method - weigh CDV*..*Naf in a bottle slightly larger than the amount of .5N NaCl/KCl solution to be added.

Equipment -
Mettler AE240, balance accurate to 0.001g (101237)
T.G. Spatulas
(21) - 15 ml Nalgene PP bottles
Versa Bath®s, Model 236 (const. temp. bath)

Reagents: 0.1 - 1.0 X_k (NaCl/KCl @ .5N) see pg 27
~9.9 gm CDV*100/200*UC*WA*HL*CPT*Naf

Procedure - 1.) Acid wash 21-15ml Nalgene PP bottles and rinse them. After rinsing, dry in oven @ 50°C until dry.
2.) Once dry, place a clean bottle on a balance, tare the balance, and then add in the given amount of CDV*..*Naf
3.) Once the weight has been recorded, tare again and ~~add in~~ pipet a specific volume of solution (10 ml in this case) and record T.G. the weight. T.G. 7-24-94

4.) Label the bottle (e.g. E_{K^+}: .01 / 0.5 N)

5.) Place all the bottles in a constant temperature water bath @ 25°C and allow the exchangeable cations between the CDV*..*Naf and .5N KCl/NaCl to come to equilibrium.

NaCl/KCl (0.5N@25°C) ISOTHERM EXPERIMENT

E_{K^+}	E_{K_z}	EXPECTED Na+ (ppm)	EXPECTED K+ (ppm)	E_i, K^+ used	Wt. Zeol. to use (gm)	Vol. Sol'n used (ml)	Wt. Zeol. used (gm)	wt. sol'n used (gm)
.01	.2		196	.1	1.037	10	1.0371	10.2195
.02	.3		391	.1	0.6144	10	.6143	10.2261
.03	.4		587	.1	0.4032	10	.4031	10.2263
.05	.51		978	.1	0.2259	10	.2259	10.2251
.075	.6		1466	.2	0.4800	10	.4801	10.2118
.09	.64		1759	.2	0.3960	10	.3960	10.2235
.1	.655		1955	.2	0.3518	10	.3518	10.2241
.13	.7		2542	.2	0.2304	10	.2306	10.2228
.15	.73		2393	.3	0.4735	10	.4737	10.2063
.18	.75		3519	.3	0.3687	10	.3686	10.2061
.2	.77		3910	.3	0.2992	10	.2991	10.2084
.24	.8		4692	.4	0.4608	10	.4606	10.2311
.3	.825		5865	.4	0.2793	10	.2794	10.2322
.35	.85		6843	.5	0.4066	10	.4067	10.1740
.4	.87		7820	.6	0.5297	10	.5295	10.1676
.5	.895		9775	.7	0.5149	10	.5150	10.2175
.6	.92		11730	.9	0.7514	10	.7517	10.2427
.7	.94		13685	1.0	0.7354	10	.7354	10.2408
~~.8~~ .75	.965		14662	1.0	0.6064	10	.6064	10.2395
.8	.96		15640	1.0	0.4800	10	.4800	10.2414
.9	.98		17595	1.0	0.2351	10	.2353	10.2436

6/25/91 T.C. 0800

Preparation of the CDV--NaCl/KCl (0.05N) Mixtures for Isotherm Experiment @ 25°C

Objective- to prepare 0.05N KCl/NaCl-CDV mixtures by adding various amounts of zeolite to specific solution volumes. zeolite being CDV*$^{100/200}$VC*WA*HL*CPT*Nat and the solution being 0.05N KCl/NaCl found from pg 31.

Method: Weigh CDV*...*Nat in a bottle slightly larger than the amount of 0.05N NaCl/KCl solution to be added.

Equipment:
- Mettler AE240, balance accurate to ±.001g (101237)
- Spatulas
- 21-30 ml Nalgene PP bottles
- Versa Bath "S", Model 236

Reagents: 0.1-1.0 X_K (NaCl/KCl@0.05N) see pg 31
for preparation
~3.1 gm CDV*$^{100/200}$VC*WA*HL*CPT*Nat

Procedure: 1.) Acid Wash 21-30 ml PP bottles, rinse them and place in oven @ 50°C until dry.
2.) Once dry, place a clean bottle on a balance, tare the balance and then add in the given amount of CDV*...*Nat
3.) Once the weight has been recorded, tare again, and pipet a specific volume of solution (25 ml in this case) and record the weight.

4.) Label the bottle (e.g. $E_K^+ = .01$ 0.05N)

5.) Place all the bottles into a constant temperature water bath @ 25°C and allow the exchangable cations between the CDV*...*Nat and 0.05N KCl/NaCl to come to equilibrium.

NaCl/KCl (0.05N@25°C) ISOTHERM EXPERIMENT

E_K^+	E_{K2}	EXPECTED Na^+(ppm)	EXPECTED K^+(ppm)	E_{i,K^+}	Wt. zeol. to use (gm)	Vol. Sol'n Used (ml)	Wt. zeol. used (gm)	Wt. Sol'n used (gm)
.01	.2		19.6	.1	.2592	25	.2592	25.0012
.02	.3		39.1	.1	.1536	25	.1535	25.0034
.03	.4		58.7	.1	.1008	25	.1007	25.0039
.05	.51		97.8	.2	.1694	25	.1695	25.0296
.075	.6		147	.3	.2160	25	.2160	25.0340
.09	.64	To 7-20-94	~~196~~ 176	.3	.1890	25	.1891	25.0489
.1	.656	7/21/94	~~254~~ 196	.3	.1759	25	.1760	25.0512
.13	.7	7/21/94	~~293~~ 254	.3	.1399	25	.1398	25.0264
.15	.73	7/21/94	~~352~~ 293	.4	.1972	25	.1972	25.0159
.18	.75	7/21/94	~~391~~ 352	.4	.1690	25	.1691	25.0090
.2	.77	7/21/94	~~464~~ 391	.4	.1496	25	.1495	25.0259
.24	.8	7/21/94	~~587~~ 464	.4	.1152	25	.1151	25.0217
.3	.825	7/21/94	~~684~~ 587	.5	.1392	25	.1396	25.0086
.35	.85		684	.6	.1684	25	.1693	25.0284
.4	.87		782	.7	.1897	25	.1897	25.0100
.5	.885		978	.8	.1931	25	.1932	25.0538
.6	.92		1173	.9	.1879	25	.1880	25.0055
.7	.94		1369	1.0	.1838	25	.1837	25.0311
.75	.95		1564	1.0	.1200	25	.1199	25.0303
.8	.96		1760	1.0	.0588	25	.0587	25.0551
.9	.98		1466	1.0	.1516	25	.1518	25.0354

6/26/91 T.6. 0800

Preparation of the CDV--NaCl/$CaCl_2$ (0.5N) mixtures for Isotherm Experiment @ 25°C

Objective - to prepare 0.5N $CaCl_2$/NaCl-CDV mixtures by adding various amounts of zeolite to specific solution volumes; the zeolite being CDV*~~HC~~*WA*HL*CDT*Naf and solution being ~~0.05~~ 0.5 N $CaCl_2$/NaCl found on pg 27.

Method - Weigh CDV*...*Naf in a bottle slightly larger than the amount of 0.5N NaCl/$CaCl_2$ solution to be added.

Equipment:
Mettler AE240, balance accurate to 0.001g (101237)
Spatulas
22 - 15ml Nalgene PP bottles
Versa Bath®S, Model 236

Reagents: 0.1 - 1.0 X_{Ca} (NaCl/$CaCl_2$ @ 0.5N) see pg 27 for preparation.
~ 16 gm CDV*HC*WA*HL*CDT*Naf

Procedure: 1.) Acid wash 22-15ml PP bottles, rinse them and place in oven @ 50°C until dry.
2.) Once dry, place a clean bottle on a balance, tare the balance and then add in the precalculated amount of CDV*...*Naf.
3.) Once the weight has been recorded, tare again, and pipet a specific volume of solution (5 & 10 ml in this case) into the bottle and record the weight.

4.) Label the bottle (e.g. E_K^+: .01 @0.5N)

5.) Place all the bottles into a constant temperature water bath @25°C and allow the exchangable cations between the CDV*...*Naf and 0.05N $CaCl_2$/NaCl to come to equilibrium.

NaCl/$CaCl_2$ (0.5N @ 25°C) ISOTHERM EXPERIMENT

E_{Ca}^{2+} T.6.	T.6. ~~E Ca~~ T.6. E_{Ca_2}	Expected Na^+ (ppm)	Expected Ca^{2+} (ppm) (T.6)	E_i Ca^{2+} used	wt. zeol. to use (gm)	Vol Soln (ml)	wt. zeol. used (gm)	wt. soln used (ml)
.02	.06		200	.1	1.536	5	1.5359	5.0823
.04	.1 T.6		401	.2	1.843	5	1.8430	5.0761
.06	.12 T.6		601	.2	1.344	5	1.3441	5.0743
.08	.16		802	.2	0.8641	5	.8639	5.0459
.1	~~.1~~ .18		1002	.3	1.280	5	1.2801	5.0876
.12	~~.225~~ .2		1202	.3	1.037	5	1.3072	5.0658
.15	~~.24~~ .225		1503 ~~2004~~	.3	.7681	5	.7680	5.0921 T.6.
.2	~~.27~~ .24		2004 ~~2505~~	.4	.9601	5	.9599	5.0879
.25	~~.29~~ .27		2585 ~~3006~~	.4	.6400	5	.6399	5.0797
.3	~~.335~~ .29 ~~.335~~		3096 ~~4008~~	.5	.7946	5	.7946	5.0937
.4	~~.385~~ .335		4008 ~~5010~~	.6	.6878	5	.6876	5.0748
.5	~~.44~~ .385		5010 ~~6012~~	.7	.5985	5	.5984	5.0895
.6	~~.49~~ .44		6012 ~~6513~~	.8	.5238	5	.5240	5.0856
.65	~~.495~~ .47		6513 ~~7014~~	.8	.3677	5	.3677	5.0645
.7	~~.525~~ .495		7014 ~~7515~~	.9	.4655	5	.4653	5.2866
.75	~~.55~~ .525		7515 ~~8016~~	.9	.3292	5	.3292	5.0771
.8	~~.58~~ .55		8016	1.0	.4189	5	.4191	5.0821
.83	~~.61~~ .58		8316	1.0	.3377	5	.3377	5.0821
.87	~~.64~~ .61		8718	1.0	.4911	10	.4911	10.2229
.9	~~.73~~ .69		9018	1.0	.3459	10	.3439	10.1954
.93	.73		9319	1.0	.2210	10	.2209	10.1918
.95	.79		9519	1.0	.1458	10	.1459	10.1948

All errors were corrected on the same day in the write-up. T.6

6/26/91 1300 T.6.

Preparation of the CDV--NaCl/$CaCl_2$ (0.05N) Mixtures for ISOTHERM EXPERIMENT @ 25°C

Objective- to prepare 0.05N NaCl/$CaCl_2$-CDV mixtures by adding various amounts of zeolite to specific solution volumes; the zeolite being CDV*100/200*NC*WA*HL*CPT*Naf and solution being 0.05N NaCl/$CaCl_2$ found on pg 31

Method- Weigh CDV*...*Naf in a bottle slightly larger than the amount of 0.05N NaCl/$CaCl_2$ solution to be added.

Equipment:
Mettler AE240, balance accurate to 0.0001g (10/237)
Spatulas
17-30ml and 3-50ml Nalgene PP bottles
Nevsa Bath®S, Model 236

Reagents- 0.1-1.0 X_{Ca} (NaCl/$CaCl_2$ @ 0.05N) see pg 31

~3.9g CDU*100/200*NC*WA*HL*CPT*Naf

Procedure- 1.) Acid Wash 17-30ml & 3-50ml PP bottles, rinse them and place in oven @ 50°C until dry.

2.) Once dry, place a clean bottle on a balance, tare the balance and then add in the previously calc amount of CDV*...*Naf.

3.) Once the weight has been recorded, tare again and pipet a specific volume of solution ((25 & 50)ml) into the bottle and record the weight.

4.) Label the bottle (e.g. $E_{Ca^{2+}}$: 0.15 / 0.05N)

5.) Place all of the bottles into a constant temperature bath @ 25°C and allow the exchangable cations between the CDV*...*Naf and 0.05N NaCl/$CaCl_2$ to come to equilibrium.

NaCl/$CaCl_2$ (0.05N @ 25°C) ISOTHERM EXPERIMENT

$E_{Ca^{2+}}$	$E_{Ca^{2+}}$	Expected Naf (ppm)	Expected Ca^{2+} (ppm)	$E_{i,Ca^{2+}}$ used	wt. zeol. to use (gm)	vol. soln used (ml)	wt. zeol. used (gm)	wt. soln used (gm)
0.015	.2		15	.1	.2448	25	.2446	25.0191
0,03	.27		30	.1	.1493	25	.1492	25.0099
0,05	.3		50	.2	.2880	25	.2879	25.0129
0,08	.34		80	.2	.2033	25	.2034	25.0159
0.1	.37		100	.2	.1557	25	.1555	25.0160
0.15	42		150	.3	2057	25	.2057	25.0063
0.2	.45		200	.4	.2560	25	.2559	25.0141
0.25	.48		251	.4	.1800	25	.1800	25.0189
0.3	.5		301	.5	.2304	25	.2304	25.0091
.4	.53		401	.6	.2174	25	.2174	25.0128
.5	.57		501	.7	.2021	25	.2022	25.0162
.55	.59		551	.7	.1465	25	.1465	25.0116
.6	.62		601	.8	.1858	25	.1858	25.0204
.65	.65		651	.9	.2323	25	.2321	25.0223
.7	.68		701	1.0	.2541	25	.2541	25.0176
.75	.72		752	1.0	.2000	25	.1999	25.0190
.8	.76		802	1.0	.1516	25	.1516	25.0144
.85	.81		852	1.0	.2134	50	.2136	50.0262
.9	.85		902	1.0	.1355	50	.1356	50.0198
.95	.9		952	1.0	.0640	50	.0640	50.0164

6/27/91 0800 T.G.

Preparation of 0.005N NaCl/KCl & 0.005N NaCl/$CaCl_2$ Solutions

Objective - To prepare various NaCl/KCl (NaCl/$CaCl_2$) solutions, but with each solution having a total normality of 0.005N.

Method - serial dilution from 0.05N solutions.

Equipment - 22-1L Nalgene PP bottles
1000 ml pipet and bulb
1000 ml volumetric flask(s)
eye dropper

Reagent - .1-1.0 X_K (X_{CaCl_2}) 0.05N KCl/NaCl ($CaCl_2$/NaCl) solutions
>25 ml deionized H_2O

Procedure - 1.) Make up 1000 ml of 0.005N KCl/NaCl ($CaCl_2$/NaCl) for each of the 0.1-1.0 X_K (X_{Ca}) solutions except make up 2000 ml of 0.005N KCl/NaCl ($CaCl_2$/NaCl) 1.0 X_K (X_{Ca}) only. Do this by pipetting 100 ml of the 0.05N solution (see pg 31) and diluting it with deionized water to 1000 ml in a volumetric flask.
NOTE: Make sure to rinse the pipet, after each use, with deionized H_2O. Dry it (partially), and rinse it with ~10 to 20 ml of (.1-1.0 X_K or .1-1.0 X_{Ca}) by pipetting the 0.05N solution into the pipet each time. Also, if

limited to only a few volumetric flasks, rinse after each solution by swirling with dist H_2O @ least 2-3 times to prevent carryover of previous solutions. The flasks do not need to be dried.
2.) Transfer the 0.005N solution into a clean, acid-wash 1000 ml PP bottle
3) Label the bottle (e.g. NaCl/KCl*.005*0.1 X_K plus date and initial)
OR
(e.g. NaCl/$CaCl_2$*.005*0.1 X_{Ca} plus date and initial)

4.) Reminder: 200 ml of the 1.0 X_K (.05N KCl/NaCl) and 1.0 X_{Ca} (.05N $CaCl_2$/NaCl) should have been diluted to 2000 ml instead of the 1000 ml that all the others became. A total of 22 L of solution should have been diluted

1L - .1 X_K	(.005N KCl/NaCl)		1L - .1 X_{Ca}	(.005N $CaCl_2$/NaCl)
1L - .2 X_K	(")		1L - .2 X_{Ca}	(")
1L - .3 X_K	(")		1L - .3 X_{Ca}	(")
1L - .4 X_K	(")		1L - .4 X_{Ca}	(")
1L - .5 X_K	(")		1L - .5 X_{Ca}	(")
1L - .6 X_K	(")		1L - .6 X_{Ca}	(")
1L - .7 X_K	(")		1L - .7 X_{Ca}	(")
1L - .8 X_K	(")		1L - .8 X_{Ca}	(")
1L - .9 X_K	(")		1L - .9 X_{Ca}	(")
2L - 1.0 X_K	(")		2L - 1.0 X_{Ca}	(")

6/28/91 0820 T.G.

Preparation of CDV -- NaCl/KCl (.005N) Mixtures for Isotherm Experiment @ 25°C

Objective - to prepare 0.005N NaCl/KCl - CDV mixtures by adding various amounts of zeolite to specific volumes of solution; the zeolite being CDV# 100/200# UC # WA # HL # CPT # Naf and solution being 0.005 N NaCl/KCl found on pg 44.

Method - Weigh CDV#...#Naf in a bottle slightly larger than the amount of .005N NaCl/KCl solution to be added.

Equipment -
Mettler AE240, balance accurate to .001g (101237)
Spatulac
16-50ml, 3-100ml, 2-250ml Nalgene PP bottles
Versa Bath S, Model 236

Reagents - 0.1 - 1.0 X_K (NaCl/KCl @ .005N) see pg 44
≈ 1.7469 gm CDV# 100/200 # UC # WA # HL # CPT # Naf

Procedure - 1.) Acid Wash 16-50ml, 3-100ml, and 2-250ml PP bottles, rinse them and place in oven @ 50°C until dry.

2.) Once dry, place a clean bottle on a balance, tare the balance and then add in the precalculated amount of CDV#...#Naf.

3.) Once the weight has been recorded, tare again and pipet a specific volume of solution (50, 100, or 250ml) into the bottle and record the weight.

4.) Label the bottle (e.g. E_{K^+} :.01 0.005N)

5.) Place all of the bottles into a constant temperature bath @ 25°C and allow the exchangable cations between the CDV#...#Naf and 0.005N NaCl/KCl to come to equilibrium.

E_{K^+}	E_{K^+}	EXPECTED Na^+ (ppm)	EXPECTED Ca^{2+} (ppm)	E_{i,K^+} used	wt zeol. to use (gm)	vol. soln used (ml)	wt. zeol. used (gm)	Wt. soln used (gm)
.01	.12		2.0	.12	.1094	50	.1093	50.0215
.02	.3		3.9	.3	.1075	50	.1077	50.0354
.03	.4		5.9	.3	.0778	50	.0777	50.0217
.05	.51		9.8	.4	.0791	50	.0790	50.0089
.075	.6		14.7	.5	.0816	50	.0817	50.0461
.09	.64		17.6	.5	.0738	50	.0738	50.0136
.1	.655		19.6	.6	.0879	50	.0880	50.0353
.13	.7		25.4	.6	.0774	50	.0774	50.0113
.15	.73		29.3	.7	.0868	50	.0867 7.6	50.0509
.18	.75		35.2	.7	.0799	50	.0799	50.0397
.2	.77		39.1	.8	.0898	50	.0899	50.0523
.24	.8		46.9	.8	.0806	50	.0806	50.0417
.3	.825		58.7	.9	.0838	50	.0838	50.0311
.35	.85		68.4	.9	.0745	50	.0745	50.0302
.4	.87		78.2	1.0	.0795	50	.0795	50.0255
.5	.895		98.8	1.0	.0645	50	.0645	50.0361
.6	.92		117	1.0	.1002	100	.1002	100.0851
.7	.94		137	1.0	.0736	100	.0736	100.0006
.75	.95		147	1.0	.0606	100	.0606	99.9732
.8	.96		156	1.0	.1202	250	.1202	248.69
.9	.98		176	1.0	.0589	250	.0589	248.99

6/28/91 1300 T.G.

Preparation of CDV-- NaCl/$CaCl_2$ (0.005N) Mixtures for Isotherm Experiment @ 25°C

Objective - to prepare 0.005N NaCl/$CaCl_2$-CDV mixtures by adding various amounts of zeolite to specific volumes of solution; the zeolite being CDV*100/200*UC*WA*HL*CPT*Naf and solution being 0.005N NaCl/$CaCl_2$ found on pg 44

Method - Weigh CDV*...*Naf in a bottle slightly larger than the amount of 0.005N NaCl/$CaCl_2$ solution to by added.

Equipment -
Mettler AE240, balance accurate to 0.001g (101237)
Spatulas
16-50ml, 4-100ml, 3-250ml Nalgene PP bottles
Versa Bath®s, Model 236

Reagents 0.1 - 1.0 X_{ca} (NaCl/$CaCl_2$ @ 0.005N) see pg 44

~ 2.6 gm CDV*100/200*UC*WA*HL*CPT*Naf

Procedure - 1.) Acid-wash 16-50ml, 4-100ml, and 3-250 PP bottles, rinse them and place in oven @ 50°C until dry.

2.) Once dry, place a clean bottle on a balance, tare the balance and then add in a precalculated amount of CDV*...*Naf.

3.) Once the weight has been recorded, tare again and pipet a specific volume of solution (50, 100, 250ml) into the bottle and record the weight.

4.) Label the bottle (e.g. $E_{Ca^{2+}}$: .01 0.005N)

5.) Place all the bottles into a constant temperature water bath @ 25°C and allow the exchangable cations between the CDV*...*Naf and 0.005N NaCl/$CaCl_2$ to come to equilibrium.

NaCl/$CaCl_2$ (0.005N @ 25°C) ISOTHERM EXPERIMENT

$E_{Ca^{2+}}$	$E_{Ca^{2+}}$	EXPECTED Na+ (ppm)	EXPECTED Ca^{2+} (ppm)	$E_{i,Ca^{2+}}$ used	Wt. Zeol to use (gm)	Vol. Sol'n used (ml)	Wt. Zeol. used (gm)	Wt. sol'n used (gm)
.01	.23		1	.3	.1453	50	.1453	50.0309
.03	.34		3	.3	.0915	50	.0916	50.0420
.05	.4		5	.4	.1008	50	.1008	50.0085
.08	.46		8	.4	.0801	50	.0802	50.0209
.1	.49		10	.5	.0940	50	.0940	50.0358
.13	.53		13	.5	.0804	50	.0806	50.0494
.17	.56		17	.6	.0801	50	.0803	50.0103
.2	.575		20	.6	.0710	50	.0711	50.0102
.23	.6		23	.7	.0902	50	.0902	50.0554
.27	.62		27	.7	.0799	50	.0800	50.0005
.3	.63		30	.8	.0914	50	.0914	50.0184
.35	.65		35	.8	.0798	50	.0798	50.0161
.4	.67		40	.9	.0860 T.G.	7/24/94 50	.0862	50.0449
.45	.685		45	.9	.0759 T.G.	7/24/94 50	.0759	50.0382
.5	.7		50	1.0	.0822 T.G.	7/24/94 50	.0822	50.0561
.6	.74		60	1.0	.0624 T.G.	7/24/94 50	.0624	50.0137
.65	.765		65	1.0	.1054 T.G.	7/24/94 100	.1055	100.0265
.7	.78		70	1.0	.0888 T.G.	7/24/94 100	.0888	100.0007
.75	.81		75	1.0	.0711	100	.0712	100.0748
.8	.83		80	1.0	.0555	100	.0555	100.0531
.85	.87		85	1.0	.0993	250	.0992	249.56
.90	.9		90	1.0	.0640	250	.0640	249.49
.95	.94		95	1.0	.0306	250	.0308	249.15

7/1/91 0800 T.G.

Prewash ~~, in an 72-60 ml~~ 7-24-94 T.G. @ least 84-15ml, 42-30ml, 72-60ml, ~~and 72-100ml~~ T.O. 7-24-94 Nalgene PP bottles for use in the 25°C Isotherm Experiment. Acid wash the bottles and then set them in an oven until dry. Cap the bottles and set aside until needed. Label bottles before use.

7/10/91 TOP

Reweigh samples CDV...*Naf*CA1 to 5 (see page 26)

Sample #	Total Weight (gm)
CDV*100/200*UC*WA*HL*CPT*Naf*CA1	16.5435
CDV* " " " " *CA2	16.5438
CDV* " " " " *CA3	16.5095
CDV* " " " " *CA4	17.4207
CDV* " " " " *CA5	16.9676

Take about 0.5g of each, put in plastic vials, label. To be sent to Texas Tech for chemical analysis.

Preparation of Zeolite Samples to be sent to Texas Tech Univ for Chemical Analysis using ICP

Method: Approximately 0.5g of the zeolite material or NBS standard is taken and placed in plastic vials. The vials are labeled A to J, 2 of the vials have NBS standards. The vial labels and the corresponding identity of the samples are given below:

A - CDV*100/200*UC*WA*HL*CPT*Naf*CA1
B - Na-feldspar; NBS-99A
C - CDV*100/200*UC*WA*HL*CPT*Naf
D - CDV*100/200*UC*WA*HL*CPT*Naf*CA2
E - CDV*100/200*UC*WA*HL*CPT*Naf*CA3
F - CDV*100/200*UC*WA*HL*CPT*Naf*CA4
G - CDV*100/200*UC*WA*HL*CPT*Naf*CA5
H - CDV*100/200*UC*WA*HL*CPT*Naf
I - CDV*100/200*UC*WA*HL*CPT*Naf
J - K-feldspar; NBS-70A

7/11/91 0800 T.G.

Once all prewashed bottles ~~had~~ been t.o. ~~are~~ dried, all the reference solutions were analyed for cations: K^+ and Na^+ in the NaCl/KCl solutions and Ca^{2+} and Na^+ in the $CaCl_2$/NaCl solutions. The references that needed to be diluted for the analysis, such as the .5N NaCl/KCl solutions were prepared in 100ml volumetrics. Below are the Reference Dilution factor, Expected reference diluted, Actual Reference diluted, and the Corrected reference from the dilutions.

NaCl / KCl (0.5N)

E_{i,K^+}	Na^+ Reference Dilution Factor	K^+	Na^+ Expected Reference diluted	K^+	Na^+ Actual Ref. Diluted	K^+	Na^+ Corrected Actual Reference.	K^+
.1	5/100	10/100	517	195	518	200	10360	~~2000~~ ~~4000~~
.2	5/100	10/100	460	391	461	400	9220	~~4000~~ ~~8000~~
.3	10/100	10/100	805	586	792	595	~~7920~~ ~~15840~~	~~4900~~ 5950
.4	10/100	10/100	690	782	686	784	~~6860~~ ~~13720~~	~~5680~~ 7840
.5	10/100	5/100	575	489	571	484	5718 ~~4680~~	~~11420~~ ~~5710~~ 9680
.6	10/100	5/100	460	586	458	581	~~4580~~ ~~4460~~	11620
.7	10/100	5/100	345	684	344	672	~~3440~~ ~~6880~~	13440
.8	—	—	—	—	—	—	—	~~3430~~
.9	10/100	5/100	115	880	117	883	1170 ~~2340~~	~~17660~~ 17660
1.0	—	5/100	—	977	-	973	—	~~19730~~ 19460

There was no Na^+ put in the $1.0\ X_{K^+}$ Reference. solutions. As for the $.8X_{K^+}$ solutions (.5N), when the procedure was optimized, Bobby Pabalan decided to use the other concentrations instead.

NaCl/KCl (0.05 ~~5~~ N)

Ref. Dilution Factor ↓

E_{i,K^+}	~~Expected Ref. Diluted~~ Na^+	~~Expected Ref. Diluted~~ K^+	Expected Reference Diluted Na^+	Expected Reference Diluted K^+	Actual Reference Diluted Na^+	Actual Reference Diluted K^+	Corrected Actual Reference Na^+	Corrected Actual Reference K^+
.1	10/100	1	103	195	106	202	1060	202
.2	1	1	920	391	922	397	922	397
.3	1	1	805	586	802	591	802	591
.4	1	1	690	782	692	770	692	770
.5	1	1	575	977	573	992	573	992
.6	1	10/100	460	117	459	116	459	1160
.7	1	10/100	345	137	349	133	349	1330
.8	1	10/100	230	156		154		1540
.9	1	10/100	115	176	116	172	116	1720
1.0	←	10/100	—	195	—	194	—	1940

NaCl/KCl (0.005 N)

E_{i,K^+}	Reference Dilution Factor Na^+	Reference Dilution Factor K^+	Expected Reference Diluted Na^+	Expected Reference Diluted K^+	Actual Reference Diluted Na^+	Actual Reference Diluted K^+	Corrected Actual Reference Na^+	Corrected Actual Reference K^+
.1	—	—	—	—	—	—	—	—
.2	1	1	92	39	92.3	37.3	92.3	37.3
.3	1	1	80	59	81.5	56.1	81.5	56.1
.4	1	1	69	78	68.8	75.5	68.8	75.5
.5	1	1	57	98	57.4	94.9	57.4	94.9
.6	1	1	46	117	45.7	108	45.1	108
.7	1	1	34	137	34	132	34	132
.8	1	1	23	156	22.5	154	22.5	154
.9	1	1	11	176	11	172	11	172
1.0	1	1	—	195	—	189	—	189

7/11/91 cont.

All the K^+ references were measured on 7/3/91 using an Ion Selective ~~Electrode~~ meter, Model 920A (Serial No. S/N 001606). 7/31/91

All the Na^+ references, including the Na^+ for the $NaCl/CaCl_2$ solutions to follow, were measured on 7/5/91 using the same Ion Selective ~~Electrode~~ meter.
TG 7-24-91

$NaCl/CaCl_2$ (0.5N)

E_i, Ca^{2+} (used)	Reference Dilution Factor		Expected Reference Diluted		Actual Reference Diluted		Corrected Actual Reference	
	Na^+	Ca^{2+}	Na^+	Ca^{2+}	Na^+	Ca^{2+}	Na^+	Ca^{2+}
0.1	5/100	10/100	517	100	520	95.3	~~10400~~	953
0.2	10/100	10/100	920	200	911	192	9110	1920
0.3	10/100	10/100	805	301	823	289	8230	2890
0.4	10/100	10/100	690	401	703	386	7030	3860
0.5	10/100	10/100	575	501	577	489	5770	4890
0.6	10/100	10/100	460	601	467	585	4670	5850
0.7	10/100	10/100	345	701	351	696	3510	6960
0.8	10/100	10/100	230	802	235	787	2350	7870
0.9	10/100	10/100	115	902	118	898	1180	8980
1.0	—	10/100	—	1002	—	1010	—	10100

$NaCl/CaCl_2$ (0.05N)

E_i, Ca^{2+} (used)	Reference Dilution Factor		Expected Reference Diluted		Actual Reference Diluted		Corrected Actual Reference	
	Na^+	Ca^{2+}	Na^+	Ca^{2+}	Na^+	Ca^{2+}	Na^+	Ca^{2+}
0.1	10/100	1	103	100	103	94.4	1030	94.4
0.2	1	1	920	200	919	195	919	195
0.3	1	1	805	301	810	296	810	296
0.4	1	1	690	401	696	391	696	391
0.5	1	1	575	501	584	481	584	481
0.6	1	1	460	601	466	585	466	585
0.7	1	1	345	701	349	693	349	693
0.8	1	1	230	802	234	813	234	813
0.9	1	1	115	902	117	905	117	905
1.0	—*	1	—	1002	—	1020	—	1020

$NaCl/CaCl_2$ (0.005N)

E_i, Ca^{2+} (used)	Reference Dilution Factor		Expected Reference Diluted		Actual Reference Diluted		Corrected Actual Reference	
	Na^+	Ca^{2+}	Na^+	Ca^{2+}	Na^+	Ca^{2+}	Na^+	Ca^+
0.1	—	—	—	—	—	—	—	—
0.2	—	—	—	—	—	—	—	—
0.3	1	1	80	30	80.8	30.7	80.8	30.7
0.4	1	1	69	40	68.5	40.7	68.5	40.7
0.5	1	1	57	50	57.1	51.2	57.1	51.2
0.6	1	1	46	60	45.5	62.2	45.5	62.2
0.7	1	1	34	70	33.6	72.6	33.6	72.6
0.8	1	1	23	80	22.0	83.0	22.0	83.0
0.9	1	1	11	90	10.6	92.4	10.6	92.4
1.0	1	1	—	100	—	102	—	102

7/11/91 cont.

All of the Ca^{2+} references were measured on 7/8/91 using an Ion Selective ~~Electrode~~ meter, Model 920A (Serial No. : S/N 001606).

7/31/91

On 7/9/91, ~~Each~~ T.G. 7-24-94 each ~~of the~~ K^+ solutions ~~were~~ was measured after the predesigned ~~solu~~ T.G. 7-24-94 dilutions were calculated. Each dilution ~~that was~~ T.G. 7-24-94 performed was done using Class A - 10 ml volumetric flasks by Paula Muller.

On 7/10/91, each ~~of the~~ T.G. 7-24-94 Na^+ solutions ~~were~~ T.G. 7-24-94 was measured after the dilutions. Each dilution was performed using Class A-10ml volumetric flasks by Paula Muller.

The results from these two days ~~follows~~ T.G. 7-24-94 are on pg 57, 58, & 59

T.G. 7/12/91 To understand how each dilution was performed, see pg 60.

NaCl/KCl (0.5N)
@ 25°C

E_{i,K^+} (used)	E_{K^+}	Dilution Factor		Expected Diluted		Actual Diluted		Corrected ~~Dilut~~ Actual	
		Na^+	K^+	Na^+	K^+	Na^+	K^+	Na^+	K^+
0.1	0.01	.5/10	1/10	569	20	574	12.9	11480	~~200~~ T.G. 7-24-94 129
0.1	0.02	.5/10	1/10	563	39	569	25.8	11380	~~390~~ T.G. 7-24-94 258
0.1	0.03	.5/10	1/10	558	59	563	52.6	11260	~~590~~ T.G. 7-24-94 526
0.1	0.05	.5/10	1/10	546	98	554	96.1	11080	~~980~~ T.G. 7-24-94 961
0.2	0.075	.5/10	1/10	532	147	532	155	10640	~~1470~~ T.G. 7-24-94 1550
0.2	0.09	.5/10	1/10	523	176	528	177	10560	~~1760~~ T.G. 7-24-94 1770
0.2	0.10	.5/10	1/10	517	196	522	198	10440	~~1960~~ T.G. 7-24-94 1980
0.2	0.13	.5/10	1/10	500	254	501	255	10020	~~2540~~ T.G. 7-24-94 2550
0.3	0.15	.5/10	1/10	489	239	488	309?	9760	~~2~~ T.G. 7-24-94 3090
0.3	0.18	.5/10	1/10	471	352	462	368	9240	3680
0.3	0.2	.5/10	1/10	460	390	458	394	9160	3940
0.4	0.24	.5/10	1/10	437	469	438	497	8760	4970
0.4	0.3	1/10	1/10	805	587	804	610	8040	6100
0.5	0.35	1/10	1/10	747	684	741	695	7410	6950
0.6	0.4	1/10	1/10	690	782	688	801	6880	8010
0.7	0.5	1/10	.5/10	575	489	567	485	5670	9700
0.9	0.6	1/10	.5/10	560	587	461	598	4610	11960
1.0	0.7	1/10	.5/10	345	684	330	687	3300	13740
1.0	0.75	1/10	.5/10	287 ~~[illegible]~~	733 ~~782~~	276	721	2760	14420
1.0	0.8	1/10	.5/10	230 ~~715~~	782 ~~880~~	219	788	2190	15760
1.0	0.9	1/10	.5/10	115 ~~287~~	880 ~~733~~	106	890	1060	17800

T.G. T.G.
7/11/91

NaCl/KCl (0.05N) @ 25°C

E_{i,K^+} (used)	E_{K^+}	Dilution Factor		Expected Diluted		Actual Diluted		Corrected Actual Measurement	
		Na^+	K^+	Na^+	K^+	Na^+	K^+	Na^+	K^+
0.1	.01	1/10	1	114	≈0	111	10	1110	100
0.1	0.02	1/10	1	113	39	114	24.3	1140	243
0.1	0.03	1/10	1	112	59	112	44.7	1120	447
0.2	0.05	1/10	1	109	98	110	87.7	1100	877
0.3	0.075	1/10	1	106	147	106	143	1060	143
0.3	0.09	1/10	1	105	176	106	187	1060	187
0.3	0.10	1/10	1	104	196	104	204	1040	204
0.3	0.13	1/10	1	100	254	100	265	1000	265
0.4	0.15	1/10	1	98	293	98	298	980	298
0.4	0.18	1/10	1	94	352	94.5	359	945	359
0.4	0.2	1/10	1	92	391	92.0	393	920	393
0.4	0.24	1/10	1	87	469	87.4	475	874	475
0.5	0.3	1/10	1	81	587	78.8	516	788	576
0.6	0.35	1/10	1	75	684	75.1	721	751	721
0.7	0.4	1/10	1	69	782	67.2	834	672	834
0.8	0.5	1/10	1/10	58	98	57.0	93.9	570	939
0.9	0.6	1/10	1/10	46	117	44.7	111	447	1110
1.0	0.7	1/10	1/10	35	137	33.3	139	333	1390
1.0	0.75	1/10	1/10	29	147	27.1	146	271	1460
1.0	0.8	1/10	1/10	23	156	22.1	157	221	1570
1.0	0.9	1/10	1/10	12	176	11.2	165	112	1650

SLOPES: : 57.2

NaCl/KCl (0.005N) @ 25°C

E_{i,K^+} (used)	E_{K^+}	Dilution Factor		Expected Diluted		Actual Diluted		Corrected Actual Measurement	
		Na^+	K^+	Na^+	K^+	Na^+	K^+	Na^+	K^+
0.2	0.01	1	1	114	2	114	1.16	114	1.16
0.3	0.02	1	1	113	4	114	2.47	114	2.47
0.3	0.03	1	1	112	6	113	4.43	113	4.43
0.4	0.05	1	1	109	10	110	8.37	110	8.37
0.5	0.075	1	1	106	15	107	14.7	107	14.7
0.5	0.09	1	1	105	18	103	18.1	103	18.1
0.6	0.10	1	1	103	20	103	21.0	103	21.0
0.6	0.13	1	1	100	25	99.2	28.4	99.2	28.4
0.7	0.15	1	1	98	29	95.7	32.8	95.7	32.8
0.7	0.18	1	1	94	35	92.7	37.4	92.7	37.4
0.8	0.2	1	1	92	39	90.5	41.8	90.5	41.8
0.8	0.24	1	1	87	47	83.9	49.5	83.9	49.5
0.9	0.3	1	1	80	59	78.1	63.1	78.1	63.1
0.9	0.35	1	1	75	68	71.6	72.6	71.6	72.6
1.0	0.4	1	1	69	78	65.3	79.6	65.3	79.6
1.0	0.5	1	1	57	98	54.8	93.4	54.8	93.4
1.0	0.6	1	1	46	117	44.2	111	44.2	111
1.0	0.7	1	1	34	137	32.9	136	32.9	136
1.0	0.75	1	1	29	147	25.5	145	25.5	145
1.0	0.8	1	1	23	156	22.0	156	22.0	156
1.0	0.9	1	1	11	176	11.0	173	11.0	173

7/11/91 1300 T.G.

Measured each NaCl/$CaCl_2$ solution using an Ion selective Electrode, Model 920A (Serial No. S/N001606). The Only Na^+ was measured. ~~Each dilution performed~~ T.G. Paula Muller performed each dilution using 10-ml Volumetric flasks. Each volumetric was washed in an acid bath followed by thorough rinsing (@ least 4 times) to ensure that there would be no solution carryover, since we had a limited number of 10-ml volumetrics. Use ~~of~~ Eppendorf pipetters ~~were used~~ since a large ~~number~~ quantity of solutions ~~needed~~ to be made. One of the eppendorfs was a constant volume (1 ml and the other was a 500 ul. Each bottle was labeled (e.g. $E_{Ca^{2+}}$ .85 .05N Na^+) The Na^+ symbolizing Na^+ ISA present

7/12/91 1600 T.G.

Measured the Ca^{2+} ions from the NaCl/$CaCl_2$ solutions using the Model 920A Ion Selective Electrode (Serial #. S/N 001606). Paula Mu performed each dilution using the proceedure listed above. Her bottles were labeled as: $E_{Ca^{2+}}$ .85 .05N Ca^{2+} where Ca^{2+} symbolized that Calcium ISA was used instead of Sodium.

7/12/91 1605 T.G.

~~NaCl/KCl~~ $CaCl_2$

The following three pages of results are from the procedures conducted on 7/11/91 and earlier this morning.

NaCl/$CaCl_2$ (0.5 N) @25°C

E_{i,Na^+} (used)	$E_{Ca^{2+}}$	Dilution Factor		Expected Diluted		Actual Diluted		Corrected Actual Measurement	
		Na^+	Ca^{2+}	Na^+	Ca^{2+}	Na^+	Ca^{2+}	Na^+	Ca^{2+}
0.1	.02	.5/10	1/10	563	20	570	23.7	11400	237
0.2	0.04	.5/10	1/10	552	40	552	49.2	11040	492
0.2	0.06	.5/10	1/10	540	60	535	66.7	10700	667
0.2	0.08	.5/10	1/10	529	80	526	95.1	10520	951
0.3	0.1	.5/10	1/10	517	100	514	121	10280	1210
0.3	0.12	.5/10	1/10	506	120	492	140	9840	1400
0.3	0.15	.5/10	1/10	489	150	485	161	9700	1610
0.4	0.2	.5/10	1/10	460	200	446	209	8920	2090
0.4	0.25	.5/10	1/10	431	251	428	243	8560	2430
0.5	0.3	.5/10	1/10	402	301	398	305	7960	3050
0.6	0.4	.5/10	1/10	345	401	341	389	6820	3890
0.7	0.5	.5/10	1/10	287	501	284	497	5680	4970
0.8	0.6	.5/10	1/10	230	601	226	590	4520	5900
0.8	0.65	.5/10	1/10	201	651	192	646	3840	6460
0.8	0.7	.5/10	1/10	172	701	174	698	3480	6980
0.9	0.75	.5/10	1/10	144	752	142	747	2840	7470
1.0	0.8	.5/10	1/10	115	802	116	814	2320	8140
1.0	0.83	.5/10	1/10	98	832	98.4	834	1968	8340
1.0	0.89	1/10	.5/10	149	456	150	423	1500	8460
1.0	0.9	1/10	.5/10	115	451	112	447	1120	8940
1.0	0.93	1/10	0.5/10	81	466	78.5	467	785	9340
1.0	0.95	1/10	0.5/10	58	476	54.7	477	547	9540
1.0									

SLOPES: - 28.8

Cont.

NaCl/$CaCl_2$ (0.05 N) @ 25°C

$E_{i,Ca^{2+}}$ (used)	$E_{Ca^{2+}}$	Dilution Factor		Expected Diluted		Actual Diluted		Corrected Actual Measurement	
		Na^+	Ca^{2+}	Na^+	Ca^{2+}	Na^+	Ca^{2+}	Na^+	Ca^{2+}
.1	.015	1/10	1	113	15	113	19.4	1130	19.4
.1	.03	1/10	1	112	30	110	36.0	1120	36.0
.2	.05	1/10	1	109	50	108	56.1	1090	56.1
.2	.08	1/10	1	106	80	97.1	81.7	1060	81.7
.2	.1	1/10	1	104	100	104	102	1040	102
.3	.15	1/10	1	95	150	98	154	950	154
.4	.2	1/10	1	92	200	89.3	203	920	203
.4	.25	1/10	1	86	251	84.9	249	860	249
.5	.3	1/10	1	81	301	79.7	295	810	295
.6	.4	1/10	1	69	401	68.4	386	690	386
.7	.5	1/10	1	58	501	58.7	488	580	488
.7	.55	1/10	1	52	551	51.8	542	520	542
.8	.6	1/10	1	46	601	46.0	587	460	587
.9	.65	1/10	1	40	651	41.2	642	400	642
1.0	.7	1/10	1	35	701	33.6	694	350	694
1.0	.75	1/10	1	29	752	27.8	744	290	744
1.0	.8	1/10	1	23	802	22.2	804	230	804
1.0	.85	1/10	1	17	852	16.3	862	170	862
1.0	.9	1/10	1	12	902	10.9	917	120	917
1.0	.95	1/10	1	6	952	5.35	953	60	953

SLOPES: 59.0 : 29.7

Cont.

NaCl/$CaCl_2$ (0.005 N) @ 25°C

$E_{i,Ca^{2+}}$ (used)	$E_{Ca^{2+}}$	Dilution Factor		Expected Diluted		Actual Diluted		Corrected Actual Measurement	
		Na^+	Ca^{2+}	Na^+	Ca^{2+}	Na^+	Ca^{2+}	Na^+	Ca^{2+}
.3	.01	1	1	114	1	113	1.89	113	1.89
.3	.03	1	1	112	3	112	3.03	112	3.03
.4	.05	1	1	109	5	107	5.73	107	5.73
.4	.08	1	1	106	8	107	7.12	107	7.12
.5	.1	1	1	103	10	104	9.84	104	9.84
.5	.13	1	1	100	13	99.9	13.1	99.9	13.1
.6	.17	1	1	95	17	94.7	18.00	94.7	18.1
.6	.2	1	1	92	20	91.2	21.0	91.2	21.0
.7	.23	1	1	89	23	90.0	21.5	90.0	21.5
.7	.27	1	1	84	27	85.2	24.6	85.2	24.6
.8	.3	1	1	80	30	82.6	27.3	82.6	27.3
.8	.35	1	1	75	35	76.7	31.4 ~~32.7~~	76.7	31.4
.9	.4	1	1	69	40	70.6	32.9	70.6	32.9
.9	.45	1	1	63	45	64.3	42.7	64.3	42.7
1.0	.5	1	1	57	50	57.8	47.9	57.8	47.9
1.0	.6	1	1	46	60	46.7	58.2	46.7	58.2
1.0	.65	1	1	40	65	40.2	63.8	40.2	63.8
1.0	.7	1	1	34	70	34.6	68.2	34.6	68.2
1.0	.75	1	1	29	75	28.6	72.3	28.6	72.3
1.0	.8	1	1	23	80	22.2	80.1	22.2	80.1
1.0	.85	1	1	17	85	16.5	838	16.5	83.8
1.0	.9	1	1	11	90	10.9	90.1	10.9	90.1
1.0	.95	1	1	6	95	5.35	94.5	5.35	94.5

SLOPES: 59.0 : 29.3

7/15/91 T.G. 1300

Measured pH of all 0.005N solutions, including references using an ion selective electrode meter, Model 920A (Serial #: SN 001606)

The purpose was to find out whether or not hydrogen was participating in the ion exchange.

T.G. 7/15/91

0.005N SAMPLES (CONTAIN CDV) see pg 46-49 for preparation

$E_{i,Ca^{2+}}$	$E_{Ca^{2+}}$	pH	temp (°C)	E_{K^+}	pH	temp (°C)	E_{i,K^+}
.3	.01	6.297	22.1	.01	6.368	23.0	T.G. .2
.3	.03	6.214	22.4	.02	6.308	22.9	.3
.4	.05	6.117	22.5	.03	6.251	22.8	.3
.4	.08	6.069	22.6	.05	6.200	23.0	.4
.5	.1	5.772	21.8	.075	6.212	22.3	.5
.5	.13	5.722	23.0	.09	6.191	22.5	.5
.6	.17	5.941	22.9	.1	6.218	22.5	.6
.6	.2	5.980	23.1	.13	6.167	22.9	.6
.7	.23	5.978	23.1	.15	6.166	22.4	.7
.7	.27	5.972	22.5	.18	6.159	22.6	.7
.8	.3	6.016	22.1	.2	6.235	22.7	.8
.8	.35	5.979	22.0	.24	6.123	22.7	.8
.9	.4	5.954	22.0	.3	6.129	22.7	.9
.9	.45	5.943	22.3	.35	6.084	23.0	.9
1.0	.5	5.880	22.3	.4	6.126	22.7	1.0
1.0	.6	5.888	22.4	.5	6.044	22.9	1.0
1.0	.65	5.913	22.3	.6	6.032	22.9	1.0
1.0	.7 .75	5.848	22.4	.7	5.965	23.0	1.0
1.0	.75 .8	5.905	22.2	.75	5.902	22.7	1.0
1.0	.8 .85	5.808	22.(	.8	5.697	23.0	1.0
1.0	.95 .9	5.714	22.3	.9	5.666	23.0	1.0
1.0	.9 .95	5.697	22.3				
1.0	.95	5.639	22.4				

0.005N References (for preparation; see pg 44)

E_{i,K^+}	pH	temp (°C)	$E_{i,Ca^{2+}}$	pH	temp (°C)
.2	5.634	23.3			
.3	5.623	23.7	.3	5.649	23.8
.4	5.625	23.9	.4	5.631	24.0
.5	5.610	23.6	.5	4.998?	23.7
.6	5.593	23.5	.6	5.634	24.0
.7	5.601	23.7	.7	5.613	24.2
.8	5.575	23.6	.8	5.618	24.0
.9	5.612	24.1	.9	5.644	24.1
1.0	5.593	23.8	1.0	5.638	24.4

7/16/91 T.G. 1030

SEM/EDS analysis was performed on the CDV * 100/200 * UC * UA * HL * CPT * Nat to check for impurities and to see if the particles had been removed from the surface of the grain and to see if any clays had formed. T.G. 7/16/91

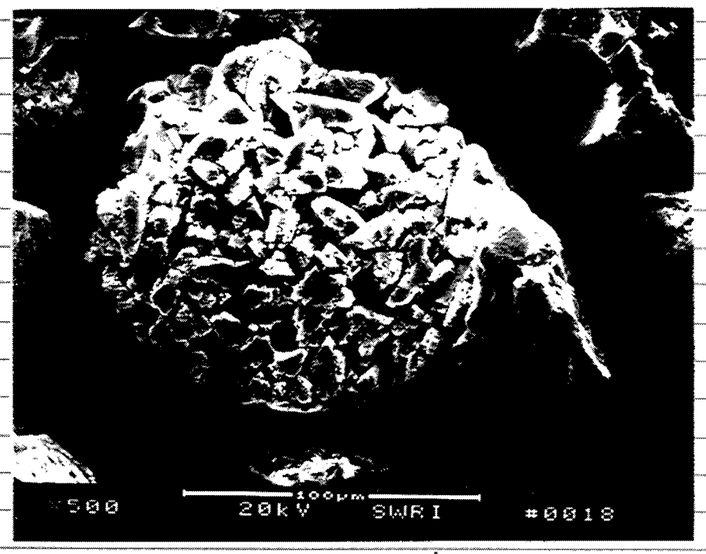
This analysis was performed using an AMRAY scanning electron microscope and Tracor Northern energy dispersive X-ray analyzer in Division 06. T.G. Harold Saldana assisted Jim Prikryl in the analysis. SEM photographs were logged into the machine notebook and the negatives were kept in Div 06 files. Xerox copies of the SEM photographs are included and originals are kept in a separate file. The photographs follows.

See pg 35 6/21/91 for more information.

7/16/91 cont.

CDV* uc* Naf

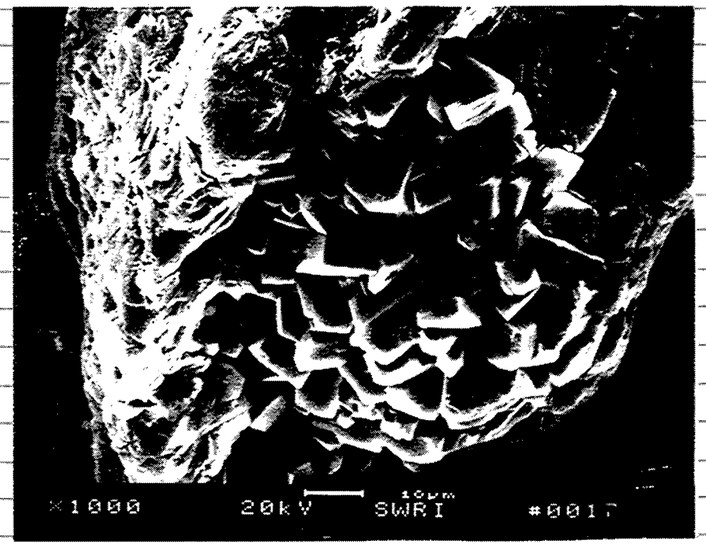
CDV* 100/200* uc* WA* HL* CPT* Naf Log# 50268



CDV* uc* Naf

CDV* 100/200* uc* WA* HL* CPT* Naf Log# 50270

7/16/91 cont.

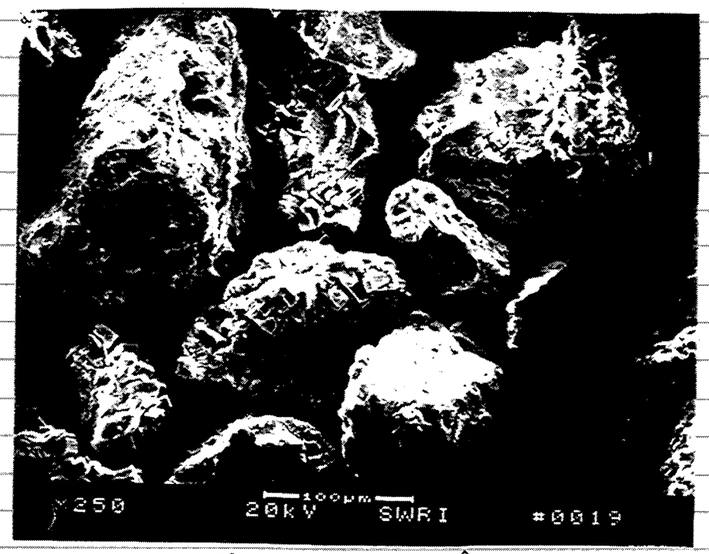


CDV* uc* Naf

CDV* 100/200* uc* WA* HL* CPT* Naf Log# 50269

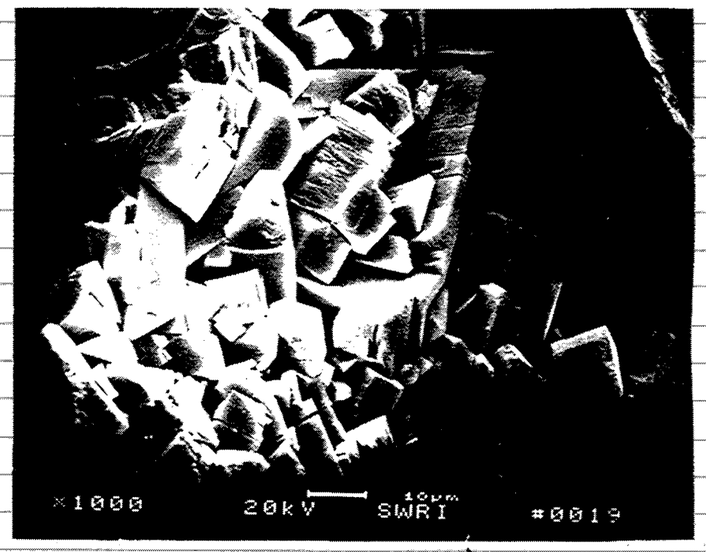
The difference between these three photographs and the next two is that these three were ultrasonically cleaned (uc) and then washed whereas the following two photographs were just washed. The purpose was to determine whether or not the washing alone removed the clays and other impurities or ultrasonic cleaning and washing was required. Since these photographs weren't returned for sometime, all the CDV* 100/200* uc* WA* HL* CPT* Naf was again ultrasonically cleaned and washed just to make sure the CDV was clean. The photographs however, show that washing alone would have done the job nicely.

7/16/91 cont.



CDV * WA * Naf

CDV * 100/200 * UC * WA * HL * CPT * Naf Log# 50271



CDV * WA * Naf

CDV * 100/200 * UC * WA * HL * CPT * Naf Log# 50272

7/17/91 1600 T.G

Preparation of the CDV-- NaCl/KCl (0.5N) Mixtures for Isotherm Experiment @ 90°C

Objective- to repeat the Ion Exchange experiment that was performed @ 25°C and observe whether or not an increase in temperature to 90°C, will cause an increase, decrease, or no change within the CDV-- NaCl/KCl (0.5N) solution.

The method, equipment, and reagents will all be exactly the same as those found on pg 36 and 37.

Procedure- 1.) Acid wash 21-15 ml Nalgene PP bottles and rinse them. After rinsing, dry in oven @ 50°C until dry.

2.) Once dry, place a clean bottle on a balance, tare the balance, and then add in the given amount of CDV * ... * Naf.

3.) Once the weight has been recorded, tare again and pipet a specific volume of solution (10 ml in this case) and record the weight.

4.) Label the bottle; side and top.

e.g. and E_{K^+} : .01
0.5N
90°C

5.) Due to the high temperature, water wasn't used in the constant temperature water bath. In its

7/17/91 cont

place high temperature Fisher Bath Oil (White Mineral Oil & Lot # 911146) having a flash point of 360°F – 400°F was used and gas masks were worn when the lids were opened.

Place the PP bottles into the 90°C oil bath and allow the exchangable cations to reach equilibrium.

NaCl/KCl (0.5N @ 90°C) ISOTHERM EXPERIMENT

E_{Kf}	E_{K2}	$E_{i,K+}$	Wt. zeol. house (g)	Vol. soln used (ml)	wt. zeol. used (g)	wt. soln used (g)
.01	.2	.1	1.037	10	1.0371	10.1751
.02	3	.1	.6144	10	.6145	10.1772
.03	.4	.1	.4032	10	.4031	10.1736
.05	.51	.1	.2259	10	.2260	10.1840
.075	.6	.2	.4800	10	.4800	10.1903
.09	.64	.2	.3960	10	.3961	10.1891
.1	.655	.2	.3518	10	.3520	10.1916
.13	.7	.2	.2304	10	.2305	10.1826
.15	.73	.3	.4735	10	.4737	10.1971
.18	.75	.3	.3687	10	.3687	10.2125
.2	.77	.3	.2992	10	.2993	10.1923
.24	.8	.4	.4608	10	.4608	10.1950
.3	.825	.4	.2793	10	.2794	10.1916
.35	.85	.5	.4066	10	.4066	10.1763
.4	.87	.6	.5297	10	.5294	10.2098
.5	.895	.7	.5149	10	.5150	10.1802
.6	.92	.9	.7514	10	.7513	10.1906
.7	.94	1.0 ~~.80~~	.7354	10	.7354	10.1973
.75	.95	1.0	~~.4800~~ .6064	10	.6063	10.1760
.8	.96	1.0	~~.2351~~ .4800	10	.4801	10.1982
.9	.98	1.0	~~.6064~~ .2351	10	.2351	10.2136

T.G. 7/17/91

7/17/91 cont

Preparation of the CDV-- NaCl/KCl (0.05N) Mixtures for Isotherm Experiment @ 90°C

Objective – to repeat the Ion Exchange experiment that was performed @ 25°C and observe whether or not an increase in temperature, to 90°C, will cause an increase shift of the cation equilibrium curve within the CDV-- NaCl/KCl (0.5N) solution.

T.G. 7-24-91

The method and equipment are the same as those found on pgs. 38 and 39

Reagents – Same as on pg 38, and White Mineral Oil Lot # 911146

Procedure – 1.) Acid wash 21 – 30ml PP bottles rinse them and place in oven @ 50° until dry.

2.) Once dry, place a clean bottle on a balance, tare the balance and then add in the given amount of CDV* ... * Wat.

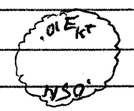
3.) Once the weight has been recorded, tare again and pipet a specific volume of solution (25 ml). T.G. 7-24-91 Record the weight.

4.) Label the bottles side and top.

e.g.

E_{Kf}: .01
T: .005 N
90°C

and



5.) Due to the high temperature white mineral oil was substituted for the water in the water bath. Preset the bath

to 90°C. Place the PP bottles into the preset bath and allow the exchangeable cations to reach equilibrium.

NaCl/KCl (0.05N @90°C) ISOTHERM EXP.

E_{K^+}	E_{K^+}	E_{i,K^+}	wt. zeol to use (g)	Vol. sol'n used (ml)	wt. zeol used (g)	wt. sol'n used (g)
.01	.2	.1	.2592	25	.2594	24.9773
.02	.3	.1	.1536	25	.1536	24.9904
.03	.4	.1	.1008	25	.1007	24.9701
.05	.51	.2	.1694	25	.1694	24.9671
.075	.6	.3	.2160	25	.2162	24.9911
.09	.64	.3	.1890	25	.1890	25.0143
.1	.655	.3	.1759	25	.1759	25.0287
.13	.7	.3	.1399	25	.1399	25.0388
.15	.73	.4	.1972	25	.1971	24.9974
.18	.75	.4	.1690	25	.1689	24.9884
.2	.77	.4	.1496	25	.1496	25.0011
.24	.8	.4	.1152	25	.1153	24.9846
.3	.825	.5	.1397	25	.1396	24.9884
.35	.85	.6	.1694	25	.1693	24.9968
.4	.87	.7	.1897	25	.1896	25.0155
.5	.895	.8	.1931	25	.1932	24.9985
.6	.92	.9	.1879	25	.1878	25.0043
.7	.94	1.0	.1838	25	.1837	24.9777
.75	.95	1.0	.1516	25	.1517	24.9776
.8	.96	1.0	.1200 ~~.0588~~ TG 7-24-94	25	.1200	24.9776
.9	.98	1.0	.0588	25	.0587	24.9817

7/18/91 1500 T.G.

Preparation of the CDV-NaCl/KCl (0.005N) Mixtures for ISOTHERM EXPERIMENT @ 90°C

Objective - to repeat the 0.005N NaCl/KCl Ion exchange experiment that was performed @ 25°C and ~~obj~~ TG 7-24-94 observe whether or not an increase in temperature, to 90°C, will cause a shift of the cation equilibrium curve within the CDV--NaCl/KCl (0.005N) solutions.

The method and equipment are the same as those found on pgs. 46 and 47.

Reagent - Same as on pg 46 and white Mineral Oil Lot #911146

Procedure - 1.) Acid wash 16-50 ml, 3-100 ml and 2-250 ml PP bottles, rinse them and place in oven @ 50°C until dry.

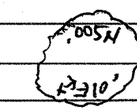
2.) Once dry, place a clean bottle on a balance, tare the balance and then add in the precalculated amount of CDV* ..* Nat.

3.) Once the weight has been recorded tare again and pipet the specific volume of solution into its respective bottle. Record the weight.

4.) Label the bottles' side and top.

e.g.

E_{K^+} : .01
.005N
90°C



5.) Due to the ~~hight~~ TG 7-24-94 high temperature, white mineral oil was substituted for the water in the water bath. Preset the bath

cont.

to 90°C. Place the PP bottles into the preset temp. bath and allow the exchangeable cations to reach equilibrium.

NaCl/KCl (0.005 N @ 90°C) ISOTHERM EXPERIMENT

E_{K+}	E_{K2}	$E_{i,K+}$	wt. zeol to use (g)	vol. ~~wt.~~ sol'n used (ml)	wt. zeol used (g)	wt. sol'n used (g)
.01	.2	.2	.1094	50	.1095	49.7240
.02	.3	.3	.1075	50	.1075	49.7728
.03	.4	.3	.0778	50	.0780	49.7704
.05	.51	.4	.0791	50	.0791	49.7630
.075	.6 TG 7-24-94	.5	.0816	50	.0817	49.8849
.09	~~.655~~ .64	.5	.0738	50	.0736	49.8449
.1	.655 TG 7-24-94	.6	.0879	50	.0878	49.7829
.13	~~.73~~ ~~.655~~ .7	.6	.0774	50	.0774	49.7909
.15	~~.75~~ .73 TG 7-24-94	.7	.0868	50	.0869	49.7952
.18	.75	.7	.0799	50	.0798	49.8528
.2	~~.8~~ .77 TG 7-24-94	.8	.0898	50	.0898	49.7892
.24	~~.825~~ .8 TG 7-24-94	.8	.0806	50	.0805	49.7991
.3	~~.85~~ .825"	.9	.0838	50	.0837	49.7589
.35	~~.87~~ .85"	.9	.0745	50	.0747	49.7760
.4	~~.895~~ .87"	1.0	.0795	50	.0795	49.8312
.5	~~.9~~ .895"	1.0	.0643	50	.0643	49.8992
.6	.92	1.0	.1003	100	.1003	99.5790
.7	.94	1.0	.0735	100	.0735	99.~~4669~~5686
.75	.95	1.0	.0607	100	.0607	99.4669
.8	.96	1.0	.1199	250	.1199	248.59
.9	.98	1.0	.0587	250	.0587	248.66

cont. 7/10/91

Preparation of CDV-- NaCl/$CaCl_2$ (0.5N) Mixtures for ISOTHERM EXPERIMENT @ 90°C

Objective - to repeat the 0.5N NaCl/$CaCl_2$ Ion exchange experiment that was performed @ 25°C and observe whether or not an increase in temperature, to 90°C, will cause a shift of the cation equilibrium curve within the CDV-- NaCl/$CaCl_2$ (0.5N) solutions.

The method and equipment are the same as those found on pgs 40 and 41

Reagents - Same as on pg 41 and White Mineral Oil Lot # 911146

Procedure: 1.) Acid wash 22-15 ml PP bottles, rinse them and place in oven @ 50°C until dry.

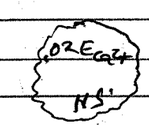
2.) Once dry, place a clean bottle on a balance, tare the balance and then add in the precalculated amount of CDV*...*Naf.

3.) Once the weight has been recorded, tare again, and pipet a specific volume of solution (5 or 10 ml) into the respective bottle. Record the weight.

4.) Label the bottles' side and top

e.g.

$E_{Ca^{2+}}$: .02
.5N
90°C



5.) Due to the high temperature, white mineral oil was substituted for the water in the water bath. Present the

cont.

bath temperature to 90°C. Place the PP bottles into the preset temp. bath and allow the exchangeable cations to reach equilibrium.

NaCl/$CaCl_2$ (0.5 N @ 90°C) ISOTHERM EXP.

$E_{Ca^{2+}}$	E_{CaZ_2}	$E_{i,Ca^{2+}}$	wt. Zeol to use (g)	Vol. Sol'n to use (ml)	wt Zeol. used (g)	wt. sol'n used (g)
.02	.06	.1	1.536	5	1.5364	5.0760
.04	.1	.2	1.843	5	1.8430	5.0785
.06	.12	.2	1.344	5	1.3442	5.0886
.08	.16	.2	.8641	5	.8640	5.0887
.1	.18	.3	1.280	5	1.2801	5.0745
.12	.2	.3	1.0370	5	1.0370	5.0694
.15	.225	.3	.7681	5	.7682	5.0820 T.G.
.2	.24	.4	.9601	5	.9602	5.0814
.25	.27	.4	.6400	5	.6399	5.0815
.3	.29	.5	.7946	5	.7946	5.0653
.4	.335	.6	.6878	5	.6877	5.0859
.5	.385	.7	.5985	5	.5985	5.0812
.6	.44	.8	.5238	5	.5238	5.1008
.65	.47	.8	.3677	5	.3678	5.1005 Pyrex
.7	.495	.9	.4655	5	.4654	5.1012 ?
.75	.525	.9	.3292	5	.3291	5.0871
.8	.55	1.0	.4189	5	.4190 T.G.	5.0925
.83	.58	1.0	.3377	5	.3378	5.0698
.87	.61	1.0	.4911	10	.4910	10.1930
.9	.67	1.0	.3439	10	.3439	10.1848
.93	.73	1.0	.2210	10	.2210	10.1976
.95	.79	1.0	.1458	10	.1459	10.1788

7/19/91 1500 T.G.

Preparation of CDV-- NaCl/$CaCl_2$ (0.05 N) Mixtures for Isotherm Experiment @ 90°C

Objective - to repeat the 0.05 NaCl/$CaCl_2$ Ion Exchange experiment that was performed @ 25°C and observe whether or not an increase in temperature, to 90°C, will cause a shift of the cation equilibrium within the CDV--NaCl/$CaCl_2$ (0.05N) solutions.

The method and equipment are the same as those found on pg 42 and 43

Reagents - Same as on pg 41 and white Mineral Oil Lot # 911146

Procedure - 1.) Acid wash 17-30 ml and 3-60 ml PP bottles, rinse them and place in oven until dry.

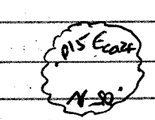
2.) Once dry, place a clean bottle on a balance, tare the balance and then add in the previously calculated amount of CDV*..*Nat.

3.) Once the weight has been recorded, tare again, and pipet a specific volume of solution (25 or 50 ml) into its respective bottle. Record the weight.

4.) Label the bottles' side and top.

e.g.

$E_{Ca^{2+}}$: .015
.05N
90°C



5.) Due to the high temperature, white mineral oil was substituted for the water in the water bath. Present the bath

temperature to 90°C. Place the PP bottles into the preset temp. bath and allow the exchangeable cations to reach equilibrium. In ~ 7 days, use an ISE meter to check the Ca^{2+} concentration.

NaCl/$CaCl_2$ (0.05N@90°C) ISOTHERM EXP.

$E_{Ca^{2+}}$	$E_{Ca^{2+}}$	E_i, Ca^{2+}	wt. salt to use (g)	vol. soln used (ml)	wt. soln used (g)	wt. soln used (g)
.015	.2	.1	.2448	25	.2448	24.9980
.03	.27	.1	.1493	25	.1494	24.9749
.05	.3	.2	.2880	25	.2878	25.0023
.08	.34	.2	.2033	25	.2033	24.9803
.1	.37	.2	.1557	25	.1558	24.9762
.15	.42	.3	.2057	25	.2058	24.9849
.2	.45	.4	.2560	25	.2561	24.9750
.28	.48	.4	.1800	25	.1799	24.9887
.3	.5	.5	.2304	25	.2304	25.0004
.4	.53	.6	.2174	25	.2175	24.9842
.5	.57	.7	.2021	25	.2021	24.9653
.55	.59	.7	.1465	25	.1464	24.9658
.6	.62	.8	.1858	25	.1858	24.9748
.65	.65	.9	.2323	25	.2324	24.9839
.7	.68	1.0	.2541	25	.2541	24.9848
.75	.72	1.0	.2000	25	.1999	24.9858
.8	.76	1.0	.1516	25	.1515	24.9850
.85	.81	1.0	.2134	50	.2134	49.9027
.9	.86	1.0	.1355	50	.1356	49.8829
.95	.9	1.0	.0640	50	.0641	49.8755

7/19/91 cont.

Preparation of CDV-- NaCl/$CaCl_2$ (0.005N) Mixtures for ISOTHERM EXPERIMENT @ 90°C

Objective - to repeat the 0.005N NaCl/$CaCl_2$ Ion Exchange experiment that was performed @ 25°C and observe whether or not an increase in temperature, to 90°C, will cause a shift of the cation equilibrium curve within the CDV-- NaCl/$CaCl_2$ (0.005N) solutions.

The Method and equipment are the same as those found on pg 48 and 49.

Reagents - Same as on pg 48 and White Mineral Oil Lot # 911146

Procedure - 1.) Acid-wash (6-50ml, 4-100, and 3-250 ml PP bottles rinse them and place in oven @ 50°C until dry.

2.) Once dry, place a clean bottle on a balance, tare the balance and then add in a precalculated amount of CDV* ...* Ref.

3.) Once the weight has been recorded, tare again and pipet a specific volume of solution (50, 100, 250 ml) into its respective bottle. Record the weight.

4.) Label the bottles' side and top e.g.

$E_{Ca^{2+}}$: .01
.005N
90°C

.01 $E_{Ca^{2+}}$ @ 300°

5.) Due to the high temperature, white mineral oil was substituted for the water in the water bath. Preset the bath

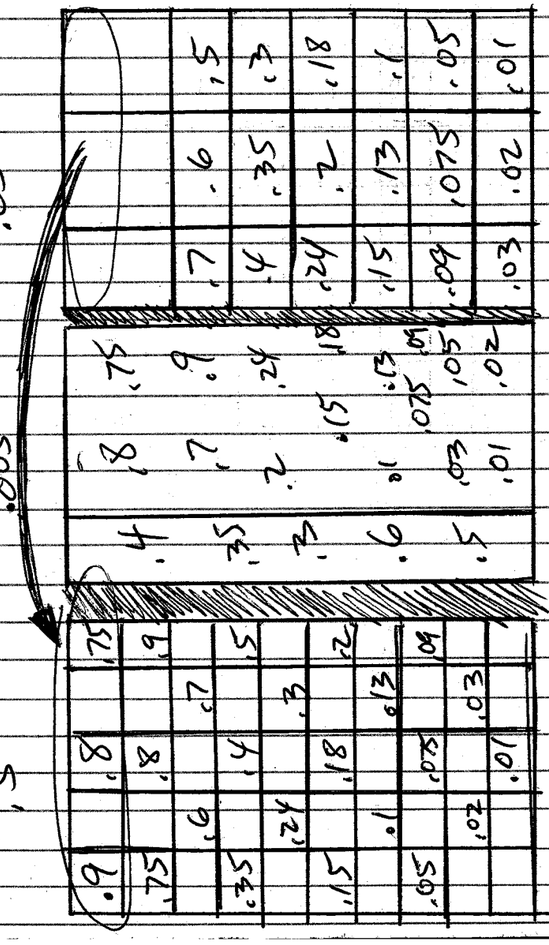
temperature to 90°C. Place the PP bottles into the preset bath and allow the exchangable cations to reach equilibrium. In ~ 7 days, use an ISE meter to check the Ca^{2+} concentration.

NaCl/$CaCl_2$ (0.005M @ 90°C) ISOTHERM EXP.

$E_{Ca^{2+}}$	E_{CaCl_2}	$E_{i,Ca^{2+}}$	wt. zeol to use (g)	Vol soln used (ml)	wt. soln used (g)	Wt. soln used (g)
.01	.23	.3	.1453	50	.1452	49.7739
.03	.34	.3	.0915	50	.0915	49.7769
.05	.4	.4	.1008	50	.1008	49.8275
.08	.46	.4	.0801	50	.0800	49.8496
.1	.49	.5	.0940	50	.0942	49.7653
.13	.53	.5	.0804	50	.0805	49.7657
.17	.56	.6	.0801	50	.0801	49.7856 T.G.
.2	.575	.6	.0710	50	.0710	49.7946
.23	.6	.7	.0904	50	.0903	49.8484
.27	.62	.7	.0799	50	.0799	49.8464
.3	.63	.8	.0914	50	.0913	49.7760
.35	.65	.9	.0798	50	.0797	49.7841
.4	.67	.9	.0860	50	.0861	49.8161
.45	.685	.9	.0757	50	.0756	49.8067
.5	.7	1.0	.0823	50	.0823	49.8120
.6	.74	1.0	.0623	50	.0623	49.7933
.65	.765	1.0	.1054	100	.1056	99.5253
.7	.78	1.0	.0886	100	.0886	99.5250
.75	.81	1.0	.0711	100	.0710	99.5391
.8	.83	1.0	.0555	100	.0557	99.5177
.85	.87	1.0	.0993	250	.0992	248.54
.9	.9	1.0	.0640	250	.0640	248.80
.95	.94	1.0	.0306	250	.0306	248.98

7/22/91 1300 T.G.

The ~~seam~~ T.G. 8/7/91 the sides and tops of all the bottles were labeled because the high temperature oil takes the marker writing off the sides. However, the oil vapors have begun removing the labels on the top of the PP bottles. Therefore, schematics have been made of all the trays in both baths. T.G.



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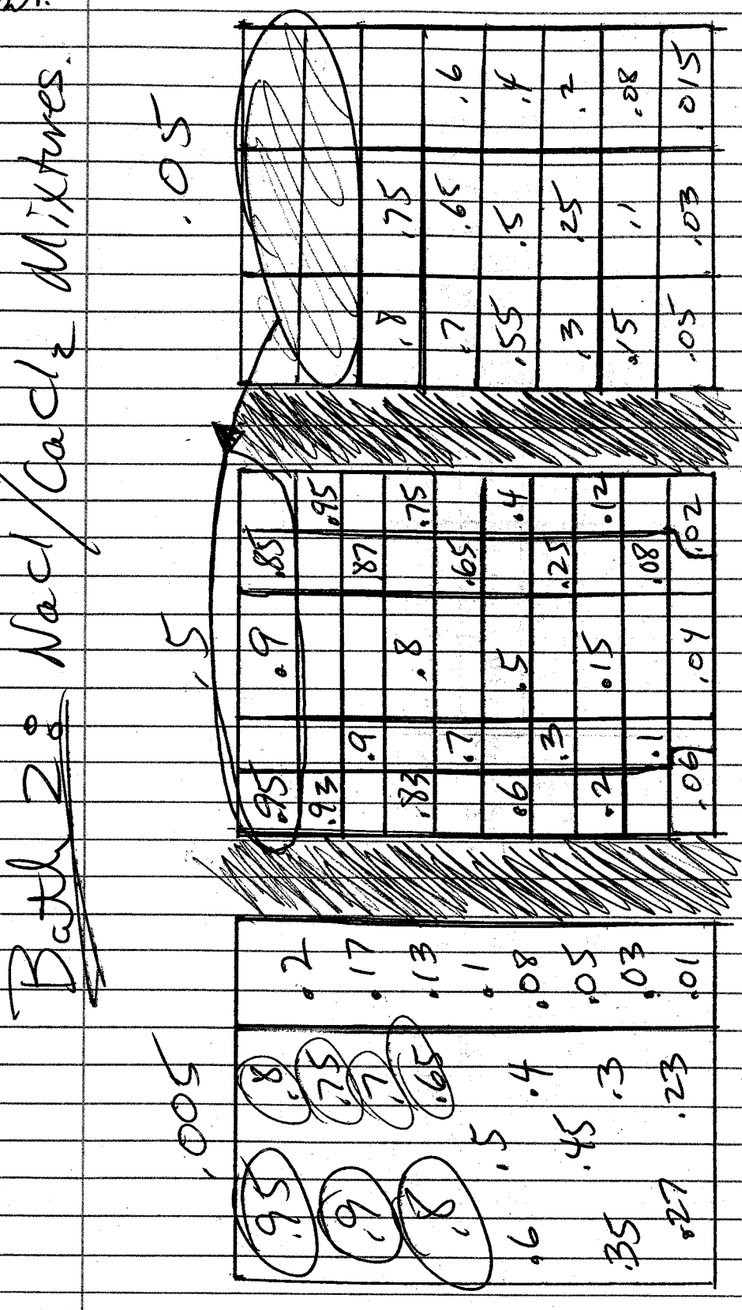


Analyses, WP — Below is a copy of analytical results from Texas Tech Univ. (See page EC-04-01)

Run for Bobby Pabalan June 9, 1991

Sample (Wt%)	A	B	C	D	E	F	G	H	I
SiO2	54.70	65.00	68.50	67.00	67.00	69.10	69.60	58.20	67.00	54.70
TiO2	0.00	0.00	0.00	0.10	0.07	0.07	0.07	0.00	0.01	0.00
Al2O3	24.54	13.90	18.14	11.10	11.19	11.33	11.47	17.40	21.13	24.30
Fe2O3^	0.04	0.16	0.10	0.04	0.50	0.47	0.47	0.02	0.00	0.04
MnO	0.00	0.02	0.00	0.02	0.01	0.00	0.00	0.00	0.00	0.00
MgO	0.02	0.73	0.05	0.37	0.20	0.26	0.26	0.05	0.03	0.02
CaO	<0.05	2.69	0.10	0.76	<0.05	0.00	0.00	7.51	2.17	<0.05
Na2O	14.50	4.23	2.56	3.53	6.47	4.04	4.02	1.92	6.41	14.20
K2O	0.02	0.10	12.21	3.55	0.47	2.00	2.00	0.20	5.31	0.04
P2O5	<0.01	0.00	0.00	0.01	0.00	0.01	0.01	0.00	0.01	<0.01
Total%	93.82	86.83	101.67	87.36	86.70	88.96	89.50	85.30	102.15	93.37
(ppm)										
Sr	7	1797	66	172	8	61	62	1606	450	8
Ba	7	3749	130	373	135	252	249	2831	2649	8
Zr	11	61	10	81	77	80	77	11	15	11
Y	<1	1	<1	15	10	10	10	<1	1	<1

Standards run with unknowns

Sample (Wt%)	J	JX	.5MHA	1.5TMS	2GSP	BCM	BCM	BCM	GSP	GSP
SiO2	68.80	69.20	30.80	91.20	140.00	55.10	56.40	56.60	67.80	69.50
TiO2	0.07	0.07	0.43	0.19	1.35	1.12	1.14	1.13	0.68	0.69
Al2O3	11.34	11.31	8.91	28.34	30.81	16.12	16.33	16.39	15.20	15.65
Fe2O3^	0.46	0.46	3.10	0.22	0.72	0.57	0.60	0.70	4.41	4.47
MnO	0.00	0.00	0.05	0.37	0.00	0.14	0.14	0.14	0.04	0.04
MgO	0.26	0.26	1.62	0.15	1.91	5.70	5.70	5.65	1.00	1.02
CaO	0.07	0.00	3.19	1.93	4.06	7.26	7.27	7.40	2.05	2.13
Na2O	4.04	4.01	2.22	13.96	5.60	4.00	4.10	4.11	2.05	2.09
K2O	2.76	2.74	0.61	6.36	11.11	2.12	2.10	2.19	5.62	5.69
P2O5	0.01	0.00	0.10	0.25	0.59	0.44	0.45	0.44	0.30	0.29
Total%	88.61	88.92	51.02	150.96	204.31	100.65	102.59	102.75	99.95	102.37
(ppm)										
Sr	56	61	201	1110	463	737	732	739	233	234
Ba	248	251	166	873	2671	920	899	927	1344	1313
Zr	80	80	76	1852	1007	134	138	139	513	513
Y	9	9	9	77	61	24	24	24	32	33

Standard Values

Sample (Wt%)	.5MHA =	BCM =	GSP =	1.5TMS =	2GSP =
SiO2	30.42	55.16	67.32	89.52	134.60
TiO2	0.43	1.11	0.66	0.18	1.32
Al2O3	8.76	15.85	15.28	27.06	30.56
Fe2O3^	3.1	8.46	4.3	8.1	8.6
MnO	0.05	0.14	0.04	0.36	0.08
MgO	1.57	5.58	0.97	0.18	1.94
CaO	3.06	7.07	2.03	1.86	4.06
Na2O	2.14	3.86	2.81	13.68	5.62
K2O	0.6	2.09	5.51	6.27	11.02
P2O5	0.09	0.45	0.28	0.23	0.56
Total%	50.22	99.77	99.28	147.44	198.36
(ppm)					
Sr	274	719	233	1116	466
Ba	153	989	1300	938	2600
Zr	72	147	500	1806	1000
Y	8	22.2	30.4	74.1	60.8

^ = Total Fe as Fe_2O_3

Na_2O, K_2O, Ba, and Sr out of standard range were run at half dilution and found to be linear.

11/30/91 Copy of results of chemical analysis of Rocky + NBS standards done by Texas Tech Univ. (see page EC-04-91).

Run for Bobby Pabalan Southwest Research Institute
Majors run 07/20/91

ICP#	983	984	986	987	989	992	993	995	996	998
Sample	A	B	C	D	E	F	G	H	I	J
(Wt%)										
SiO2	68.88	66.67	67.65	67.64	67.69	67.82	67.61	67.49	68.50	68.86
TiO2	0.07	0.01	0.06	0.06	0.06	0.06	0.06	0.06	0.06	0.01
Al2O3	11.38	20.84	11.20	11.22	11.18	11.23	11.23	11.26	11.35	18.98
Fe2O3^	0.43	0.07	0.41	0.42	0.42	0.41	0.42	0.40	0.41	0.09
MnO	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00
MgO	0.20	0.04	0.17	0.19	0.18	0.17	0.17	0.17	0.17	0.05
CaO	0.03	2.15	0.01	0.01	0.01	0.01	0.01	0.01	0.01	0.13
Na2O	5.67	6.33	6.32	5.84	6.06	6.31	6.24	6.32	6.36	2.55
K2O	1.67	5.31	0.66	1.25	0.94	0.64	0.62	0.57	0.66	11.92
P2O5	0.01	0.01	0.00	0.01	0.01	0.00	0.01	0.00	0.00	0.00
Total%	88.32	101.43	86.48	86.64	86.55	86.66	86.36	86.28	87.53	102.58
(ppm)										
Sr	30	442	10	20	14	10	10	9	10	67
Ba	202	2578	134	179	160	137	131	131	134	124
Zr	82	33	83	81	83	78	81	80	80	12
Y	9	1	10	9	9	10	9	9	9	0

Run for Bobby Pabalan Southwest Research Institute
Trace run 07/24/91

ICP#	1020	1021	1033	1034	1037	1038	1042	1043	1045	1046
Sample	A	B	C	D	E	F	G	H	I	J
(ppm)										
Rb	73	113	40	62	52	41	40	40	40	530
Sr	28	445	8	18	12	8	8	8	8	69
Ba	195	2682	132	176	163	141	143	141	145	134
Zr	73	7	77	75	73	75	72	75	77	1
Y	10	2	10	10	10	10	10	10	10	1
Sc	1.3	0.1	1.2	1.3	1.2	1.2	1.2	1.3	1.3	0.1
V	1	<1	2	1	2	1	4	2	3	1
Cr	2	2	2	2	1	1	3	0	2	1
Ni	3	5	2	2	<1	1	<1	2	1	0
Cu	7	3	1	1	1	1	0	0	7	3
Zn	9	4	7	7	7	6	6	6	7	2
Nb	15	<1	15	14	17	17	17	16	15	1

Standard values used in calibrations

Standard==>	.5MHA	ABA	BCM	TMS	MHA	GSP-1	RGM-T	1.5TMS	2GSP
Source==>>	TTU	TTU	TTU	TTU	TTU	USGS	TTU	TTU	TTU
(Wt%)									
SiO2	30.42	45.80	55.16	59.68	68.84	67.32	72.87	89.52	134.64
TiO2	0.43	2.09	1.11	0.12	0.85	0.66	0.27	0.18	1.32
Al2O3	8.76	14.36	15.85	18.04	17.51	15.28	13.61	27.06	38.56
Fe2O3*	3.10	12.27	8.46	5.40	6.20	4.30	1.85	8.10	8.60
MnO	0.05	0.19	0.14	0.24	0.10	0.04	0.04	0.36	0.08
MgO	1.57	11.34	5.48	0.12	3.13	0.97	0.30	0.18	1.94
CaO	3.06	10.61	7.07	1.24	6.11	2.03	1.25	1.86	4.06
Na2O	2.14	3.05	3.86	9.12	4.28	2.81	4.10	13.68	5.62
K2O	0.60	1.41	2.09	4.18	1.19	5.51	4.24	6.27	11.02
P2O5	0.09	0.46	0.45	0.15	0.18	0.28	0.07	0.23	0.56
Total**	50.20	101.58	99.67	98.29	108.39	99.20	98.60	147.44	198.40
(ppm)									
Rb	9.5	27	34	120	19	254	150	180	508
Sr	274	537	719	744	548	233	110	1116	466
Ba	153	404	909	620	306	1300	893	930	2600
Zr	72.35	153.1	146.6	1204	144.7	500	222.7	1806	1000
Y	8	29.7	22.2	49.4	16	30.4	26.1	74.1	60.8
Sc	6.45	29.06	18.7	0.5	12.9	6.6	4.6	0.75	13.2
V	51	219.9	137	0	102	54	11.7	0	100
Cr	24.3	404.1	157	4.6	48.6	12	1.7	6.9	24
Ni	17.35	250	93.7	4.6	34.7	12	0.8	6.9	24
Cu	9.2	49.8	46.4		18.4	33			66
Zn	32.5	88	72	224	65	98	32	336	196
Nb	5.6	47.7	34.1	332	11.2	29	10.8	498	58

Standards run as samples

ICP#	1000	982	988	999	994	1001	985	997	991
Sample	.5MHA	1.5TMS	2GSP	2GSP	BCM	GSP	GSP	MHA	RGMT
(Wt%)									
SiO2	30.68	91.02	137.00	136.00	55.88	67.93	67.71	61.39	73.99
TiO2	0.42	0.19	1.33	1.34	1.12	0.68	0.68	0.85	0.28
Al2O3	8.89	27.60	38.21	38.35	15.99	15.10	15.17	17.78	13.76
Fe2O3^	3.06	8.13	8.71	8.81	8.63	4.43	4.42	6.16	1.91
MnO	0.05	0.36	0.08	0.08	0.14	0.04	0.04	0.10	0.04
MgO	1.54	0.15	1.88	1.91	5.50	0.98	0.99	3.09	0.31
CaO	3.08	1.89	3.94	4.06	7.13	2.04	2.01	6.09	1.25
Na2O	2.18	13.67	5.58	5.62	3.92	2.82	2.79	4.32	4.11
K2O	0.59	6.30	10.82	10.53	2.06	5.18	5.36	1.18	4.20
P2O5	0.09	0.24	0.59	0.56	0.41	0.29	0.30	0.18	0.04
Total%	50.58	149.55	200.14	199.27	100.78	99.48	99.47	101.14	99.88
(ppm)									
Sr	276	1108	464	467	719	231	229	549	107
Ba	158	886	2624	2603	905	1318	1333	316	845
Zr	69	1512	1023	1017	141	508	509	128	198
Y	8.19	75.7	60.3	60	24	30.4	31	18.2	27.7

Standards run as samples

ICP# Sample (ppm)	1022 .5MHA	1044 .5MHA	1039 1.5TMS	1048 2GSP	1036 ABA	1026 BCM	1035 BLANK	1047 BLANK	1018 MHA	1025 MHA	1019 RGMT	Average Blank
Rb	9.1	9.1	180		27	34	0	-0.3	19.6	18.5	150	-0.1
Sr	270	295	1136	470	559	721	-0	-0	557	547	110	-0
Ba	148	161	946	2719	425	899	-8	-8	312	302	883	-8
Zr	61	67	6324	959	156	214	3	3	133	186	242	3
Y	9	9	75	58	30	23	-1	-1	18	18	27	-1
Sc	6.8	7.1	0.7	11.9	29.0	19.6	0.2	0.2	13.5	13.5	5.0	0.2
V	54	59	3	103	225	143	3	4	104	106	14	3
Cr	23	23	7	23	401	162	2	2	44	45	3	2
Ni	16	17	6	13	258	99	2	2	31	30	4	2
Cu	6	7	5	73	54	49	2	2	12	12	9	2
Zn	36	33	307	183	75	72	2	3	66	62	34	3
Nb	5	7	532	64	46	35	3	2	9	11	12	3

7/31/91 T.G. 1600 T.G. 7.24.94

~~The~~ Purpose: The solutions in the baths see pg 81 & 82, must be drawn directly from the bath to ensure that no ion exchange occurs as a result of cooling when out of the bath.

Procedure: On 7/23/91, enough of each solution was drawn out of the PP bottles, while they were in the 90°C oil bath, and ~~labeled~~ T.G. 7-24-94 identically labeled. For example; a total of 1.5 ml is required of the 0.09 E_{K^+} solution in order to prepare 10 ml for Na^+ measurement and 10 ml for K^+ measurement by dilution. Therefore, draw off ~~80~~ T.G. 7-24-94 no less than 2 ml of the solution and place in an identically labeled bottle. Do this by using 5 or 10 cc syringes, 25 mm syringe filter holder, and biomedical pipetting needles to ensure that T.G. 7-24-94 no CDV goes into the syringe. ~~If~~ Note: If CDV does make it into the syringe, it

may cause further ion exchange upon cooling. Repeat for T.G. all 63 bottles, and allow them to cool. Dilute the solutions accordingly.

To 7-24-94 Solutions that need diluting should be diluted to 10ml in class A volumetric flasks. Use Eppendorf pipetters since a large quantity of solutions need to be made. e.g. One of the Eppendorfs was a constant volume 1ml and the other was a 500 ul. Label each bottle. e.g. The bottles for this particular isotherm experiment were labeled as:

E_{K^+} : .3 .5N 90°C → E_{K^+} : .3 .5N 90°C Na^+ ; E_{K^+} : .3 .5N 90°C K^+

where Na^+ & K^+ represent the respective ISA which will be ~~placed~~ T.G. 7-24-94 added to the bottles for measurement.

8/1/91 0800 T.G. NaCl/KCl

On 7/26/91, all ~~K~~ T.G. 7-24-94 solutions were measured for K^+.

On 7/30/91 and 7/31/91 all NaCl/KCl and NaCl/$CaCl_2$ solutions were measured for the Na^+ concentration.

For preparation of the NaCl/$CaCl_2$ solutions, see pg 86. These dilutions were performed on 7/29/91.

The results follow. The ion concentrations

cont.

were measured using an Ion Selective meter, Model 920A (Serial Nos: ~~00~~ S/N 001606).

NaCl/KCl (0.5N)
@ 90°C

E_{i,K^+} (used)	E_{K^+}	Dilution Factor		Actual Diluted		Corrected Measurement	
		Na^+	K^+	Na^+	K^+	Na^+	K^+
0.01	.01	.5/10	1/10	615	31.9	12300	319
0.1	.02	.5/10	1/10	607	54.6	12140	546
0.1	.03	.5/10	1/10	627	86.7	12540	867
0.1	.05	.5/10	1/10	567	124	11340	1240
0.2	.075	.5/10	1/10	558	214	11160	2140
0.2	.09	.5/10	1/10	540	237	10800	2370
0.2	.1	.5/10	1/10	536	242	10720	2420
0.2	.13	.5/10	1/10	521	309	10420	3090
0.3	.15	.5/10	1/10	510	368	10200	3680
0.3	.18	.5/10	1/10	513	438	10260	4380
0.3	.2	.5/	1/10	507	490	10140	4900
0.4	.24	.5/10	1/10	468	583	9360	5830
0.4	.3	1/10	1/10	859	688	8590	6880
0.5	.35	1/10	1/10	776	760	7760	7600
0.6	.4	1/10	1/10	795	1000	7950	10000
0.7	.5	1/10	.5/10	583	518	5830	10360
0.9	.6	1/10	.5/10	462	686	4620	13720
1.0	.7	1/10	.5/10	338	760	3380	15200
1.0	.75	1/10	.5/10	273	773	2730	15460
1.0	.8	1/10	.5/10	219	856	2190	17120
1.0	.9	1/10	.5/10	125	1100	1250	22000

Avg. slope of K^+ electrode: 57.2 mV
Avg slope of Na^+ electrode: 57.3 mV

cont. NaCl/KCl (0.05N)
@ 90°C

E_{i,K^+} (used)	E_{K^+}	Actual Diluted Factor		Dilution Factor		Corrected Measurement	
		Na^+	K^+	Na^+	K^+	Na^+	K^+
0.1	0.01	138	27.9	1/10	1	1380	27.9
0.1	.02	140	48.5	1/10	1	1400	48.5
0.1	.03	158	93.8	1/10	1	1580	93.8
0.2	.05	133	128	1/10	1	1330	128
0.3	.075	126	186	1/10	1	1260	186
0.3	.09	131	240	1/10	1	1310	240
0.3	.1	127	237	1/10	1	1270	237
0.3	.13	122	308	1/10	1	1220	308
0.4	.15	120	352	1/10	1	1200	952
0.4	.18	111	400	1/10	1	1110	400
0.4	.2	117	436	1/10	1	1170	436
0.4	.24	109	500	1/10	1	1090	500
0.5	.3	102	635	1/10	1	1020	635
0.6	.35	100	721	1/10	1	1000	721
0.7	.4	84.5	806	1/10	1	845	806
0.8	.5	72.5	94.3	1/10	1/10	725	943
0.9	.6	59.5	121	1/10	1/10	595	1210
1.0	.7	45.5	134	1/10	1/10	455	1340
1.0	.75	41.3	152	1/10	1/10	413	1520
1.0	.8	36.0	152	1/10	1/10	360	1520
1.0	.9	23.7	166	1/10	1/10	237	1660

Avg. slope of K^+ electrode: 57.2 mV
Avg. slope of Na^+ electrode: 57.5 mV

cont. NaCl/KCl (0.005 N) @ 90°C

E_{i,K^+} (used)	E_{K^+}	Dilution Factor		Actual Measurement		Corrected Measurement	
		Na^+	K^+	Na^+	K^+	Na^+	K^+
0.2	.01	1	1	114	3.54	114	3.54
0.3	.02	1	1	115	5.41	115	5.41
0.3	03	1	1	116	9.58	116	9.58
0.4	.05	1	1	114	16.2	114	16.2
0.5	.075	1	1	102	25.4	102	25.4
0.5	.09	1	1	101	29.7	101	29.7
0.6	.1	1	1	97.9	31.2	97.9	31.2
0.6	.13	1	1	106	36.3	106	36.3
0.7	.15	1	1	~~94.0~~	40.4	103	40.4
0.7	.18	1	1	~~103~~	46.5	94	46.5
0.8	.2	1	1	88.4	51.1	88.4	51.1
0.8	.24	1	1	80.1	59.4	80.1	59.4
0.9	.3	1	1	77.2	70.6	77.2	70.6
0.9	.35	1	1	70.3	81.5	70.3	81.5
1.0	.4	1	1	64.8	89.9	64.8	89.9
1.0	.5	1	1	52.9	104	52.9	104
1.0	.6	1	1	44.2	130	44.2	130
1.0	.7	1	1	34.4	146	34.4	146
1.0	.75	1	1	29.9	160	29.9	160
1.0	.8	1	1	22.1	160	22.1	160
1.0	~~.85~~	1	1	10.9	195	10.9	195

Avg. slope of K^+ electrode: 57.9 mV
Avg. slope of Na^+ electrode: 56.4 mV

cont NaCl/$CaCl_2$ (0.05 N) @ 90°C

$E_{i,Ca^{2+}}$ (used)	$E_{Ca^{2+}}$	Dilution factor		Actual Measurement		Corrected Measurement	
		Na^+	Ca^{2+}	Na^+	Ca^{2+}	Na^+	Ca^{2+}
.1	.015	1/10	1	140	7.09	1400	7.09
.1	.03	1/10	1	126	16.8	1260	16.8
.2	.05	1/10	1	124	21.4	1240	21.4
.2	.08	1/10	1	119	46.1	1190	46.1
.2	.1	1/10	1	121	64.3	1240	64.3
.3	.15	1/10	1	121	110	1210	110
.4	.2	1/10	1	114	152	1140	152
.4	.25	1/10	1	107	212	1070	212
.5	.3	1/10	1	98.1	255	981	255
.6	.4	1/10	1	849	356	849	356
.7	.5	1/10	1	732	466	732	466
.7	.55	1/10	1	599	515	599	515
.8	.6	1/10	1	59.6	563	596	563
.9	.65	1/10	1	54.3	605	543	605
1.0	.7	1/10	1	45.0	678	450	678
1.0	.75	1/10	1	36.1	750	361	750
1.0	.8	1/10	1	28.8	808	288	808
1.0	.85	1/10	1	21.6	900	216	900
1.0	.9	1/10	1	14.1	900	141	900
1.0	.95	1/10	1	6.67	992	66.7	992

Avg slope of Ca^{2+} electrode: 28.9
Avg slope of Na^+ electrode: 59.4

cont.

NaCl/$CaCl_2$ (0.005N) @90°C

$E_{i,Ca^{2+}}$ (used)	$E_{Ca^{2+}}$	Dilution Factor		Actual Measurement		Corrected Measurement	
		Na^+	Ca^{2+}	Na^+	Ca^{2+}	Na^+	Ca^{2+}
0.3	.01	1	1	125	.0780	125	.0780
0.3	.03	1	1	131	.257	131	.257
0.4	.05	1	1	121	.453	121	.453
0.4	.08	1	1	126	1.43	126	1.43
0.5	.1	1	1	125	1.94	125	1.94
0.5	.13	1	1	122	4.34	122	4.34
0.6	.17	1	1	107	9.62	107	9.62
0.6	.2	1	1	106	13.7	106	13.7
0.7	.23	1	1	111	12.2	111	12.2
0.7	.27	1	1	105	17.5	105	17.5
0.8	.3	1	1	103	19.5	103	19.5
0.8	.35	1	1	101	27.1	101	27.1
0.9	.4	1	1	96.0	34.6	96.0	34.6
0.9	.45	1	1	76.8	36.5	76.8	36.5
1.0	.5	1	1	75.7	44.6	75.7	44.6
1.0	.6	1	1	56.3	55.9	56.3	55.9
1.0	.65	1	1	45.6	62.3	45.6	62.3
1.0	.7	1	1	38.5	69.8	38.5	69.8
1.0	.85	1	1	25.0	81.8	35.0	81.8
1.0	.8	1	1	25.5	82.4	25.5	82.4
1.0	.85	1	1	17.0	84.0	17.0	84.0
1.0	.9	1	1	11.3	89.6	11.3	89.6
1.0	.95	1	1	5.70	94.7	5.70	94.7

8/1/91 1100 T.G.

The Ca^{2+} ions were measured earlier in the day and the data are on the previous two pages.
No isotherm was constructed of the .5N NaCl/$CaCl_2$ solutions because too much evaporation occured in the bottles when the lids came loose from the heat. Some evaporation probably also occured in the 0.05 & 0.005N NaCl/$CaCl_2$ solutions as well, but they were analyzed anyways.

If evaporation occured in the NaCl/KCl solutions, it wasn't noticed.

Reasons: The average NaCl/KCl solution remained in the water bath for 6 days, whereas the NaCl/$CaCl_2$ solutions were in for nearly 10 days.

8/2/91 0800 T.G.

On 7/23/91 and 7/29/91, the NaCl/KCl & NaCl/$CaCl_2$ solutions were diluted, respectively. The rpm's were turned to 0 and the bath was cut back on to keep the solutions @ a constant temperature. The temperature was recorded for each solution as the mixtures were drawn from the pp. bottles.
The temperatures follow on the next two pgs.
Note: They weren't put in earlier because I was simply recording them to make sure that

Cont.

The temperature was remaining the same. ~~Once~~ T.G. Since they were, I didn't feel that it was necessary to put them in. However, here they are anyways.

.5N NaCl/KCl			.05N NaCl/KCl			.005N NaCl/KCl	
E_{K^+}	temp.(°C)		E_{K^+}	temp.(°C)		E_{K^+}	temp.(°C)
.01	89.9		.01	89.2		.01	89.7
.02	90.5		.02	89.1		.02	90.4
.03	90.0		.03	89.8		.03	90.7
.05	90.4		.05	90.3		.05	90.4
.075	89.8		.075	89.8		.075	90.6
.09	90.3		.09	90.3		.09	90.3
.1	90.2		.1	90.0		.1	89.7
.13	89.9		.13	89.8		.13	89.8
.15	90.5		.15	89.8		.15	90.6
.18	90.4		.18	90.2		.18	90.3
.2	90.0		.2	90.0		.2	90.5
.24	89.7		.24	89.8		.24	90.5
.3	90.2		.3	89.9		.3	89.7
.35	90.2		.35	89.9		.35	90.0
.4	90.5		.4	90.4		.4	89.8
.5	90.4		.5	90.2		.5	89.9
.6	89.7		.6	90.4		.6	90.0
.7	89.7		.7	90.4		.7	90.0
.75	89.9		.75	90.5		.85	89.9
.8	90.1		.8	90.4		.8	90.0
.9	90.4		.9	90.3		.9	90.6

Cont.

.5N NaCl/$CaCl_2$			.05N NaCl/$CaCl_2$			.005N NaCl/$CaCl_2$	
$E_{Ca^{2+}}$	temp(°C)		$E_{Ca^{2+}}$	temp(°C)		$E_{Ca^{2+}}$	temp(°C)
.02	NONE		.015	90.5		.01	90.5
.04	"		.03	90.6		.03	89.9
.06	"		.05	90.3		.05	89.8
.08	"		.08	89.8		.08	90.5
.1	"		.1	90.4		.1	90.5
.12	"		.15	90.2		.13	90.4
.15	"		.2	89.7		.17	89.9
.2	"		.25	90.0		.2	89.7
.25	"		.3	90.4		.23	90.4
.3	"		.4	90.4		.27	90.3
.4	"		.5	90.1		.3	89.8
.5	"		.55	89.8		.35	90.4
.6	"		.6	90.2		.4	90.3
.65	"		.65	90.5		.45	90.2
.7	"		.7	90.4		.5	90.5
.75	"		.75	89.7		.6	90.5
.8	"		.8	89.8		.65	89.9
.83	"		.85	90.1		.7	90.5
.87	"		.9	89.8		.75	90.5
.9	"		.95	89.9		.8	90.2
.93	"		—	—		.85	89.8
.95	"		—	—		.9	89.8
—	←		—	—		.95	90.4

Recall that no .5N NaCl/$CaCl_2$ samples were drawn because of the evaporation that occurred in more than 1/4 of the bottles.

8/2/91 1000 T.O.

Remeasured all NaCl/$CaCl_2$ solutions from the 25°C isotherm experiment, for Ca^{2+} ion concentration.

Purpose: To check the reproducibility of my results.

NaCl/$CaCl_2$ (@25°C)

0.5N		0.05 N		0.005	
$E_{Ca^{2+}}$	Ca^{2+} ions (ppm)	$E_{Ca^{2+}}$	Ca^{2+} ions (ppm)	$E_{Ca^{2+}}$	Ca^{2+} ions (ppm)
.02	22.4	.015	17.0	.01	1.98
.04	46.6	.03	32.5	.03	3.10
.06	62.4	.05	51.8	.05	5.90
.08	92.5	.08	74.2	.08	7.28
.1	115	.1	94.0	.1	9.83
.12	135	.15	142	.13	13.2
.15	162	.2	184	.17	17.7
.2	208	.25	228	.2	21.2
.25	242	.3	269	.23	21.5
.3	293	.4	359	.27	25.2
.4	393	.5	453	.3	28.4
.5	485	.55	495	.35	32.9
.6	584	.6	550	.4	31.0
.65	626	.65	591	.45	42.7
.7	684	.7	657	.5	49.1
.75	741	.75	712	.6	58.8
.8	778	.8	766	.65	63.8
.83	794	.85	804	.7	67.7
.87	408	.9	861	.75	72.9
.9	443	.95	915	.8	79.1
.93	449			.85	84.5
.95	453			.9	90.0
				.95	92.6

cont.

Average slope of .5N Ca^{2+} electrode: 28.8 mV
Average slope of 0.05N Ca^{2+} electrode: 28.8 mV
Average slope of 0.005N Ca^{2+} electrode: 29.5 mV

NaCl/$CaCl_2$ References (@25°C)

0.5N		0.05 N		0.005N	
$E_{i,Ca^{2+}}$	Ca^{2+} ions (ppm)	$E_{i,Ca^{2+}}$	Ca^{2+} ions (ppm)	$E_{i,Ca^{2+}}$	Ca^{2+} ions (ppm)
0.1	88.1	0.1	98.8	0.1	—
0.2	182	0.2	197	0.2	—
0.3	276	0.3	290	0.3	30.2
0.4	368	0.4	386	0.4	40.1
0.5	472	0.5	481	0.5	49.6
0.6	550	0.6	576	0.6	60.6
0.7	651	0.7	671	0.7	69.3
0.8	759	0.8	790	0.8	79.3
0.9	868	0.9	879	0.9	898
1.0	969	1.0	980	1.0	96.4

Average slope of 0.5N References: 29.5
Average slope of 0.05N References: 29.5
Average slope of 0.005N Ref.: 29.8

NOTE: None of these results have been multiplied by their respective dilution factor.

T.G. 12/23/91

98 — See pg 168 for Na^+ analyses of NaCl/KCl sol'ns.

8/5/91 0800 T.G.

Remeasured all NaCl/KCl solutions from the 25°C isotherm experiment.
Purpose: To check the reproducibility of earlier K^+ measurements.

NaCl/KCl (@ 25°C)

0.5N		0.05N		0.005N	
E_{K^+}	K^+ ions (ppm)	E_{K^+}	K^+ ions (ppm)	E_{K^+}	K^+ ions (ppm)
.01	10.4	.01	9.38	.01	.894
.02	22.3	.02	24.0	.02	2.30
.03	48.3	.03	45.2	.03	4.49
.05	94.9	.05	85.1	.05	8.33
.075	142	.075	142	.075	14.4
.09	162	.09	179	.09	18.0
.1	185	.1	188	.1	20.5
.13	248	.13	243	.13	27.8
.15	293	.15	308	.15	31.8
.18	359	.18	360	.18	36.8
.2	383	.2	384	.2	41.0
.24	464	.24	486	.24	51.7
.3	555	.3	601	.3	60.0
.35	674	.35	705	.35	71.8
.4	729	.4	797	.4	77.1
.5	456	.5	92.6	.5	99.9
.6	576	.6	116	.6	120
.7	666	.7	136	.7	135
.75	726	.75	146	.75	149
.8	768	.8	154	.8	156
.9	895	.9	165	.9	165

cont. Average slope of 0.5N K^+ electrode: 57.0
Average slope of 0.05N K^+ electrode: 57.2
Average slope of 0.005N K^+ electrode: 58.0.

NaCl/KCl References (@ 25°C)

0.5N		0.05N		0.005N	
E_{i,K^+}	K^+ ions (ppm)	E_{i,K^+}	K^+ ions (ppm)	E_{i,K^+}	K^+ ions (ppm)
0.1	180	0.1	196	0.1	~~36.3~~ —
0.2	361	0.2	383	0.2	8/5/91 ~~54.1~~ 36.3
0.3	563	0.3	572	0.3	T.G. ~~71.9~~ ~~71.9~~ ~~54.~~
0.4	755	0.4	776	0.4	71.9 ~~92.2~~ ~~92.2~~ ~~71.~~
0.5	461	0.5	981	0.5	92.2 ~~115~~ ~~115~~
0.6	564	0.6	114	0.6	~~1~~ 115
0.7	640	0.7	133	0.7	132
0.8	—	0.8	151	0.8	150
0.9	870	0.9	172	0.9	167
1.0	958	1.0	195	1.0	186

Average slope of 0.5N References: 56.6
Average slope of 0.05N References: 57.5
Average slope of 0.005N References: 57.9

Note: None of these results have been multiplied by their respective dilution factor.

8/7/91 1200 T.G.

Preparation of the CDV-- NaCl/KCl (0.5N) Mixtures for ISOTHERM Experiment @ 80°C

Everything except the temperature, is the same as that found on pg 69.
NOTE: Keep the lids tight to prevent evaporation.

NaCl/KCl (0.5N @ 80°C) ISOTHERM EXPERIMENT

E_{K+}	E_{K2}	$E_{i,K+}$	wt. zeol to use (g)	Vol. soln used (ml)	wt. zeol used (g)	wt. soln used (g)
.01	.2	.1	1.037	10	1.0373	10.1801
.02	.3	.1	.6144	10	.6144	10.1771
.03	.4	.1	.4032	10	.4032	10.1773
.05	.51	.1	.2259	10	.2258	10.1862
.075	.6	.2	.4800	10	.4799	10.1780
.09	.64	.2	.3960	10	.3960	10.1767
.1	.655	.2	.3518	10	.3517	10.1804 T.G.
.13	.7	.2	.2304	10	.2304	10.1884 T.G.
.15	.73	.3	.4735	10	.4737	10.1988
.18	.75	.3	.3687	10	.3687	10.1895
.2	.77	.3	.2992	10	.2993	10.2020
.24	.8	.4	.4608	10	.4608	10.1888
.3	.825	.4	.2793	10	.2792	10.1782
.35	.85	.5	.4066	10	.4065	10.1864
.4	.87	.6	.5297	10	.5297	10.1869
.5	.895	.7	.5149	10	.5149	10.1746
.6	.92	.9	.7514	10	.7513	10.1920
.7	.94	1.0	.7354	10	.7353	10.2044
.75	.95	1.0	~~.4800~~ .6064	10	.6065	10.2117
.8	.96	1.0	~~.2357~~ .4800	10	.4801	10.2153
.9	.98	1.0	~~.6064~~ T.G. .2351	10	.2352	10.2052

cont.

Preparation of the CDV-- NaCl/KCl (0.05N) Mixtures for Isotherm Experiment @ 80°C

Everything, except the temperature, is the same as that found on pg ~~69~~ 71. T.G. 7-24-94
NOTE: Keep the lids tight to prevent evaporation.

NaCl/KCl (0.05N @ 80°C) ISOTHERM EXPERIMENT

E_{K+}	E_{K2}	$E_{i,K+}$	wt. zeol to use (g)	Vol. soln used (ml)	wt. zeol used (g)	wt. soln used (g)
.01	.2	.1	.2592	25	.2591	24.9676
.02	.3	.1	.1536	25	.1535	24.9699
.03	.4	.1	.1008	25	.1008	24.9742
.05	.51	.2	.1694	25	.1694	24.9730
.075	.6	.3	.2160	25	.2159	24.9969
.09	.64	.3	.1890	25	.1889	24.9778
.1	.655	.3	.1759	25	.1759	25.0045
.13	.7	.3	.1399	25	.1398	24.9935
.15	.73	.4	.1972	25	.1971	24.9872
.18	.75	.4	.1690	25	.1689	24.9775
.2	.77	.4	.1496	25	.1495	24.9940
.24	.8	.4	.1152	25	.1153	24.9874
.3	.825	.5	.1397	25	.1397	24.9990
.35	.85	.6	.1694	25	.1693	25.0003
.4	.87	.7	.1897	25	.1898	24.9986
.5	.895	.8	.1931	25	.1931	24.9789
.6	.92	.9	.1879	25	.1880	25.0085
.7	.94	1.0	.1838	25	.1838	24.9735
.75	.95	1.0	.1516	25	.1516	24.9778
.8	.96	1.0	.1200	25	.1200	24.9737
.9	.98	1.0	.0588	25	.0589 T.G.	24.9904

8/17/91 1630 T.G.

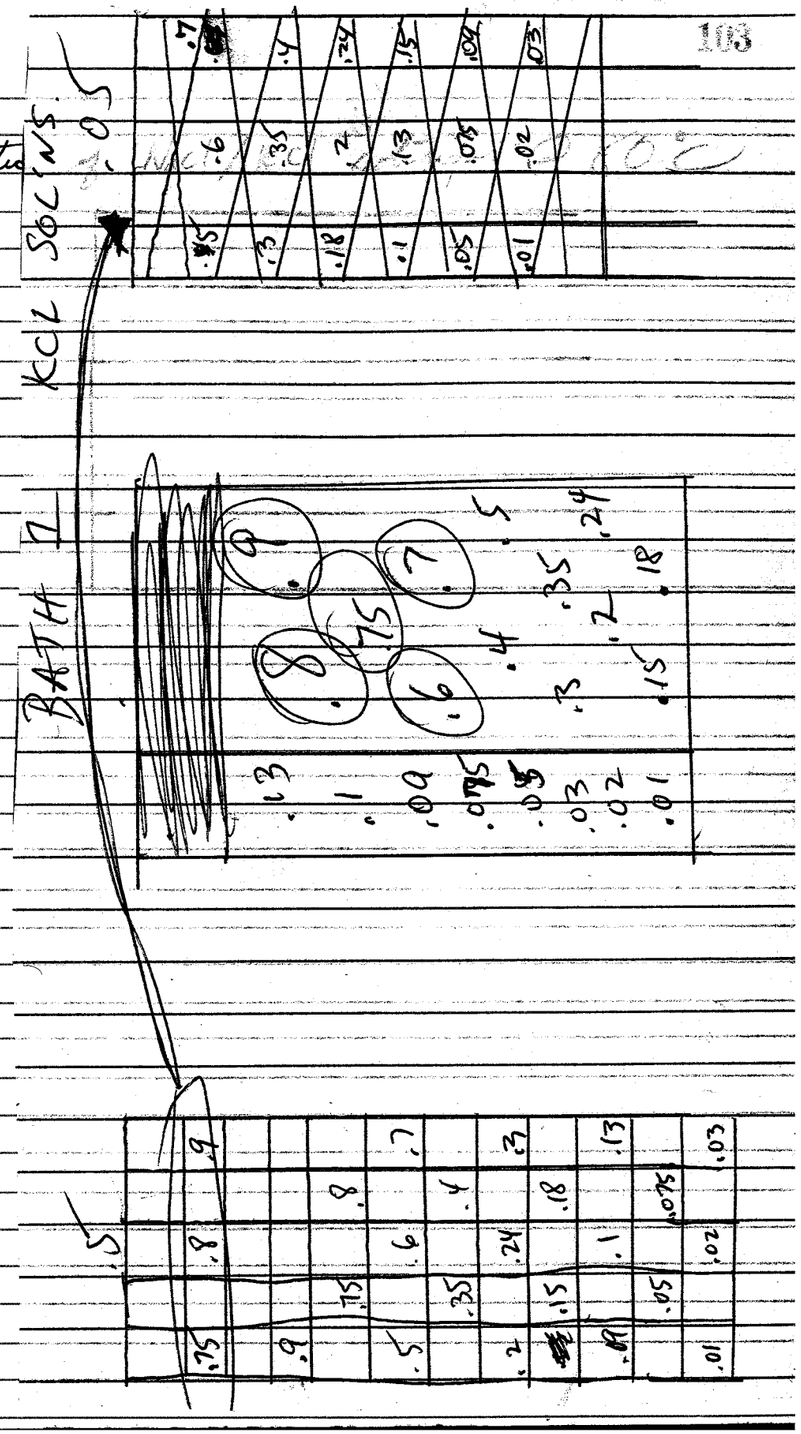
Preparation of the CDV -- NaCl/KCl - (0.005N) Mixtures for ISOTHERM EXPERIMENT @ 80°C

Everything, except the temperature, is the same as that found on pg 73.
NOTE: keep the lids tight to prevent evaporation

NaCl/KCl (0.005N @ 80°C) ISOTHERM EXPERIMENT

E_{K+}	E_{K2}	T.G. ~~wt. zeol to use (g)~~ E_{i,K^+}	wt. zeol. to use (g)	Vol. sol'n used (ml)	wt. zeol. used (g)	wt. sol'n used (g)
.01	.2	.2	.1094	50	.1093	49.9311
.02	.3	.3	.1075	50	.1074	49.8388
.03	.4	.3	.0778	50	.0777	49.9111
.05	.51	.4	.0791	50	.0790	49.9584
.075	.6	.5	.0816	50	.0816	49.8329
.09	.64	.5	.0738	50	.0738	49.8634
.1	.655	.6	.0879	50	.0880	49.8333
.13	.7	.6	.0774	50	.0774	49.8286
.15	.73	.7	.0868	50	.0867	49.8683
.18	.75	.7	.0799	50	.0798	49.8464
.2	.77	.8	.0898	50	.0899	49.8311
.24	.8	.8	.0806	50	.0805	49.8329
.3	.825	.9	.0838	50	.0837	49.8395
.35	.85	.9	.0745	50	.0744	49.8381
.4	.87	1.0	.07945	50	.0794	49.8455
.5	.895	1.0	.0644	50	.0644	49.8804
.6	.92	1.0	.1002	100	.1003	99.6148
.7	.94	1.0	.0735	100	.0734	99.5862
.75	.95	1.0	.0606	100	.0605	99.5826
.8	.96	1.0	.1200	250	.1199	248.49
.9	.98	1.0	.0588	250	.0590	248.52

cont. Schematic



T.6 12/23/91
See pg 168 for Na^+ analyses of NaCl/KCl sol'ns.

8/6/91 1600 T.C

Remeasured all NaCl/$CaCl_2$ solutions from the 25°C isothermal experiment.

PURPOSE: To check the reproducibility of earlier Na^+ measurements.

NaCl/$CaCl_2$ (@ 25°C)

0.5 N		0.05 N		0.005 N	
$E_{Ca^{2+}}$	Na^+ ions (ppm)	$E_{Ca^{2+}}$	Na^+ ions (ppm)	$E_{Ca^{2+}}$	Na^+ ions (ppm)
.02	565	.015	113	.01	114
.04	545	.03	111	.03	113
.06	530	.05	109	.05	107
.08	526	.08	103	.08	109
.1	508	.1	97.3	.1	105
.12	486	.15	96.9	.13	100
.15	479	.2	90.9	.17	95.2
.2	443	.25	84.4	.2	91.3
.25	421	.3	79.8	.23	90.6
.3	396	.4	68.7	.27	85.5
.4	339	.5	57.7	.3	83.0
.5	279	.55	51.8	.35	76.7
.6	222	.6	46.3	.4	70.1
.65	191	.65	41.6	.45	65.3
.7	169	.7	33.7	.5	57.8
.75	141	.75	27.5	.6	46.5
.8	115	.8	21.9	.65	40.1 T.6.
.83	97.6	.85	15.9	.7	34.4
.87	148	.9	10.9	.75	28.6
.9	112	.95	5.23	.8	22.1
.93	76.8			.85	16.4
.95	53.7			.9	10.8
				.95	5.57

cont. Average slope of 0.5N Na^+ electrode: 58.5
Average slope of 0.05N Na^+ electrode: 58.8
Average slope of 0.005N Na^+ electrode: 58.8

NaCl/$CaCl_2$ References (@ 25°C)

0.5 N		0.05 N		0.005 N	
$E_{i,Ca^{2+}}$	Na^+ ions (ppm)	$E_{i,Ca^{2+}}$	Na^+ ions (ppm)	$E_{i,Ca^{2+}}$	Na^+ ions (ppm)
0.1	526	0.1	106	0.1	—
0.2	943	0.2	950	0.2	—
0.3	822	0.3	828	0.3	80.1
0.4	714	0.4	708	0.4	69.5
0.5	598	0.5	587	0.5	58.1
0.6	475	0.6	466	0.6	46.3
0.7	355	0.7	349	0.7	34.9
0.8	241	0.8	237	0.8	23.2
0.9	121	0.9	118	0.9	11.7
1.0	—	1.0	—	1.0	—

Average slope of the 0.5N References: 58.8
Average slope of the 0.05N References: 58.8
Average slope of the 0.005N References: 58.8

NOTE: None of these results have been multiplied by their respective dilution factor.

8/9/91 0900hrs JP

Recovery of Clinoptilolite (CDV*100/200*UC*WA*HL*CPT*Naf) used in 90°C Ion Exchange Experiments.

Objective - recover CDV*...*Naf used in 90°C ion exchange experiments.

Method - remove CDV*...*Naf from sample bottles then wash, filter, and dry.

Equipment -
a) filter paper
b) beakers, funnels
c) Mettler AE240 balance accurate to 0.1mg (SN-101237)
d) Blue M drying oven (SN-OV3-70344.

Procedure -
a) CDV*100/200*UC*WA*HL*CPT*Naf used in 90°C ion exchange experiments was washed out of sample bottles and collected by filtering.

b) Collected CDV*...*Naf was washed with acetone and then water.

c) The CDV*...*Naf was dryed in an oven at ~60°C.

d) The dryed CDV*...*Naf was placed in a glass beaker and labeled:
CDV*100/200*UC*WA*HL*CPT*Naf*REC.

e) Weight of CDV*...*REC + container = 86.42g

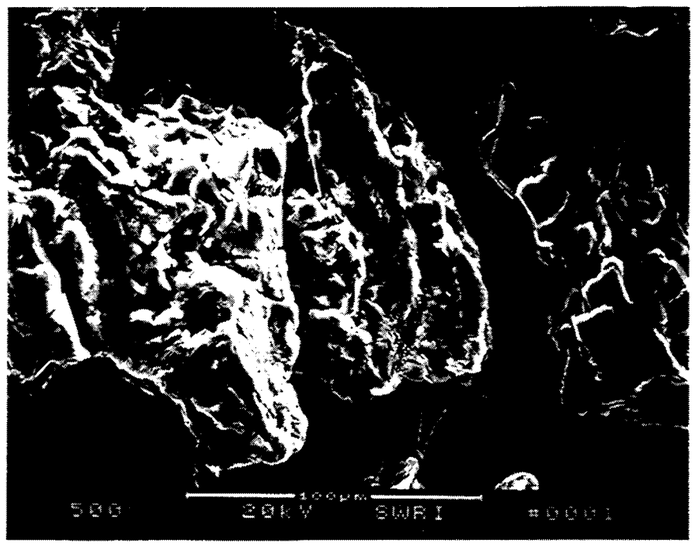
8/12/91 0900 hrs JP

SEM analysis was performed on sample CDV*100/200*UC*WA*HL*CPT*Naf*REC to check for secondary mineral formation and/or dissolution features. The analysis was done using an AMRAY scanning electron microscope in Div. 06. Harold Saldana of Div. 06 assisted in the analysis. Mike Leahy of DIV 15 carbon coated the samples. SEM photographs were logged into the machine notebook & negatives were kept in Div. 06 files. Xerox copies of the SEM photographs are included and originals are kept in a separate notebook.

Two separate samples of CDV*...*REC were analyzed with the SEM. Samples were prepared by mixing graphite powder and duco cement and smearing this substance on an aluminum stub. CDV*...*REC powder was then sprinkled onto the substance and allowed to dry.

SEM photographs follow:

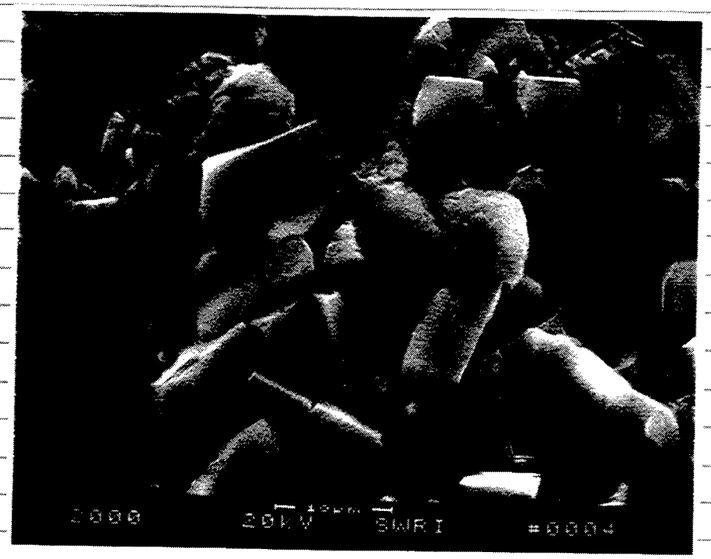
Log# 51173



log# 51174

Log # 51175

Log # 51176



Log# 51177

Log# 51178

In general the recovered clinoptilolite (CDV*...*REC) appears similar to clinoptilolite (CDV*...*NAS) not used in the ion exchange experiment. However, some dissolution features, such as etching, are evident. Secondary mineral formation is not readily apparent in the photos.

1/10/91 1500 T.G.

Abstract:
After the NaCl/KCl solutions have remained in the oil bath @ 80°C, for three full days, ~~then were~~ T.G. 7-24-94 enough of each solution was drawn off and allowed to cool in respectively labeled PP bottles and then diluted for analyses.

Purpose: The NaCl/KCl solutions in the bath, see pg 103, must be drawn directly from the bath to ensure that no ion exchange occurs as a result of cooling when out of the bath.

Equipment:
63 - 30 ml PP bottles
T.G. 8/1/91 ~~65~~ 5 to 10 cc syringes
T.G. 8/1/91 ~~65~~ - 25 mm syringe filter holder
Biomedical pipetting needle
Rubber gloves
Gas mask w/ ORGANIC FILTER
T.G. 8/1/91 10 ml Class A Volumetric Flasks.

Procedure: 1.) Draw off @ least 3 ml for the 0.5 N solutions and place each solution in its respective bottle.
Use the 5 cc syringes for this.
Clean and dry the filters and needles if necessary.
2.) Draw off @ least 18 ml for the 0.05 N solutions and place each in its respective bottle. Use 10 cc syringes.
3.) Using cleaned filters and needles, pipette T.G. 7-24-94 off @ least 28 ml

cont.
of the 0.005 ml solutions and place each in its respective bottle.
Allow the bottles to cool and T.G. 7-24-94 dilute each solutions predesigned volume to 10 ml for analyses.

5.) Once the bottles have cooled solutions that need diluting should be diluted in Class A 10 ml volumetric flasks.
Use constant volume eppendorf pipetters.
NOTE: For this experiment, each of the eppendorf's pipet tip was used only once. Each tip was rinsed with 17.7 MΩ-cm water to ensure that all the solution left the tip. In other words, leave the tip in the top of the 10-ml volumetric and rinse with ~2 ml into the flask.

6.) Fill each volumetric to 10-ml line. Pour the solutions into respectively labeled PP bottles.
e.g. The bottles for this particular isotherm experiment were labeled as:

E_{K^+}: .03
.5 N
80°C

→

E_{K^+}: .03	E_{K^+}: .03
.5 N	.5 N
80°C Na^+	80°C K^+

where Na^+ and K^+ represent the respective ISA which will be added to the bottles for measurement.

Note: for this particular solution, .5 ml was diluted to 10 for Na^+ measurement and 1 ml was diluted to 10 for K^+ measurement.

8/12/91 1200 T.G.

Preparation of the CDV-- NaCl/$CaCl_2$ (0.5N) Mixtures for Isotherm Experiment @ 80°C

Everything, except the temperature, is the same as that found pg 75.
Note: Keep the lids tight to prevent evaporation

NaCl/$CaCl_2$ (0.5N @ 80°C) ISOTHERM EXP.

$E_{Ca^{2+}}$	E_{CaZ_2}	$E_{i,Ca^{2+}}$	wt. zeol to use (g)	vol sol'n used (ml)	wt. zeol used (g)	wt. sol'n used (g)
.02	.06	.1	1.536	5	1.5359	5.0797
.04	.1	.2	1.843	5	1.8431	5.0902
.06	.12	.2	1.344	5	1.3439	5.0785
.08	.16	.2	0.8641	5	.8641	5.0870
.1	.18	.3	T.G. 1.280	5	1.2801	5.0811
.12	.2	.3	1.037	5	1.0372	5.0861
.15	.225	.3	0.7681	5	.7681	5.0859
.2	.24	.4	0.9601	5	.9601	5.0765
.25	.27	.4	0.6400	5	.6402	5.0767
.3	.29	.5	0.7946	5	.7946	5.0833
.4	.335	.6	0.6878	5	.6877	5.0927
.5	.385	.7	0.5985	5	.5985	5.0923
.6	.44	.8	0.5238	5	.5238	5.0889
.65	.47	.8	0.3677	5	.3677	5.0972
.7	.495	.9	0.4655	5	.4655	5.0827
.75	.525	.9	0.3292	5	.3292	5.0912
.8	.55	1.0	0.4189	5	.4189	5.0970
.83	.58	1.0	0.3377	5	.3376	5.0948
.87	.61	1.0	0.4911	10	.4910	10.2053
.9	.67	1.0	0.3439	10	.3438	10.2057
.93	.73	1.0	0.2210	10	.2209	10.2026
.95	.79	1.0	0.1458	10	.1457	10.2052

cont.

Preparation of the CDV-- NaCl/$CaCl_2$ (0.05N) Mixtures for Isotherm Experiment @ 80°C

Everything, except the temperature, is the same as that found on pg 77.
Note: Keep the lids tight to prevent evaporation.

NaCl/$CaCl_2$ (0.05N @ 80°C) ISOTHERM EXP.

$E_{Ca^{2+}}$	E_{CaZ_2}	$E_{i,Ca^{2+}}$	wt. zeol to use (g)	Vol sol'n used (ml)	wt. zeol. used (g)	wt sol'n used (g)
.015	.2	.1	.2448	25	.2448	24.9841
.03	.27	.1	.1493	25	.1493	24.9801
.05	.3	.2	.2880	25	.2879	25.0006
.08	.34	.2	.2033	25	.2032	24.9965
.1	.37	.2	.1557	25	T.24.94 B Tea .1556 ~~.20~~	24.9931
.15	.42	.3	.2057	25	.2057	24.9986
.2	.45	.4	.2560	25	.2560	24.9895
.25	.48	.4	.1800	25	.1801	24.9878
.3	.5	.5	.2304	25	.2304	24.9763
.4	.53	.6	.2174	25	.2174	24.9851
.5	.57	.7	.2021	25	.2023	24.9802
.55	.59	.7	.1465	25	.1464	25.0013
.6	.62	.8	.1858	25	.1859	24.9815
.65	.65	.9	.2323	25	.2323	24.9876
.7	.68	1.0	.2541	25	.2540	24.9860
.75	.72	1.0	.2000	25	.2001	24.9939
.8	.76	1.0	.1516	25	.1518	25.0070
.85	.81	1.0	.2134	50	.2134	49.9638
.9	.85	1.0	.1355	50	.1354	49.9621
.95	.9	1.0	.0640	50	.0641	49.9525

8/12/91 1300 T.G.

Preparation of the COV-- NaCl/$CaCl_2$ (0.005N) Mixtures for ISOTHERM ~~Experiment~~ @ 80°C

Everything, except the temperature, is the same as that found on pg 79.

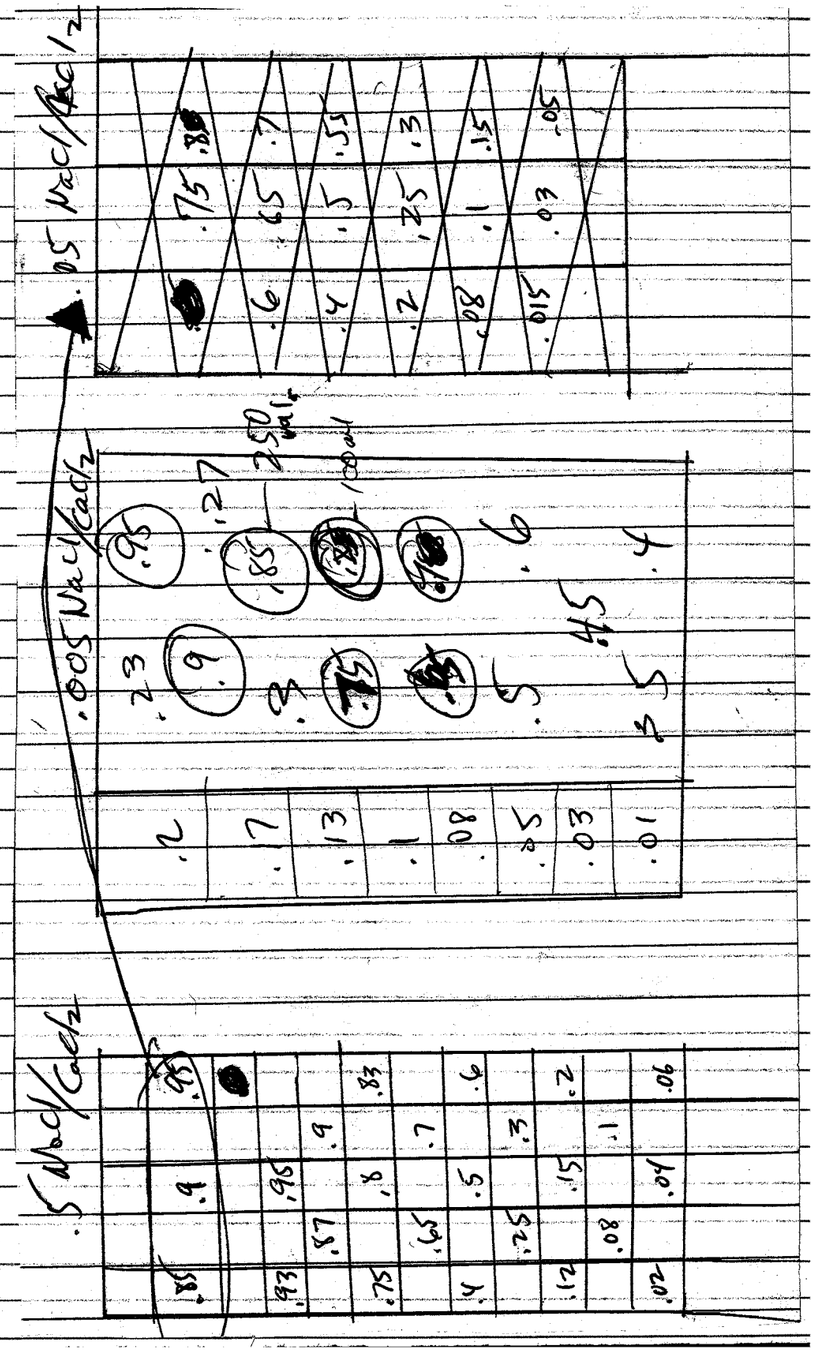
Note: keep the lids tight to prevent evaporation

NaCl/$CaCl_2$ (0.005N @ 80°C) ISOTHERM EXP.

E_{Cl^-}	E_{CaCl_2}	$E_{i,ca^{++}}$	wt. rec'd to use (g)	Vol soln used (ml)	wt. rec'd used (g)	wt. soln used (g)
.01	.23	.3	.1453	50	.1452	49.8196
.03	.34	.3	.0915	50	.0916	49.8233
.05	.4	.4	.1008	50	.1008	49.8111
.08	.46	.4	.0801	50	.0800	49.8123
.1	.49	.5	.0940	50	.0941	49.8139
.13	.53	.5	.0804	50	.0803	49.8119
.17	.56	.6	.0801	50	.0800 T.G.	49.8268
.2	.575	.6	.0710	50	.0710	49.8406
.23	.6	.7	.0902	50	.0902	49.8330
.27	.62	.7	.0799	50	.0799	49.8327
.3	.63	.8	.0914	50	.0913	49.8249
.35	.65	.8	.0798	50	.0797	49.8271
.4	.67	.9	.0860	50	.0859	49.8385
.45	.685	.9	.0757	50	.0756	49.8854 T.G.
.5	.7	1.0	.0823	50	.0822	49.9550
.6	.74	1.0	.0623	50	.0623	49.9458
.65	.765	1.0	.1054	100	.1055	99.6371 T.G. 99.6241
.7	.78	1.0	.0886	100	.0885	99.6195 T.G. 8/21/91
.75	.81	1.0	.0711	100	.0711	99.6401
.8	.83	1.0	.0555	100	.0557	248.77
.85	.87	1.0	.0993	250	.0991	248.68
.9	.9	1.0	.0640	250	.0640	248.59
.95	.94	1.0	.0306	250	.0305	

cont.

SCHEMATIC OF NaCl/$CaCl_2$ @ 80°C



8/14/91 1100 T-6.

Preparation of K^+ Standards

Objective - To prepare various K^+ standards used in calibrating the Ion Selective Meter.

Method - Weigh an original amount of KCl into a container and dilute to 1000 ml. A serial dilution was not used.

Equipment -
Mettler AE240, balance accurate to 0.001g (10123)
Spatula and 1000 and 2000 ml Volumetrics.
Pipets (Class A 50 ml, 25ml, 10ml, 5ml, 1ml, .3ml)

Reagents -
Potassium Chloride (Lot #885967)
@ least 9L of 17 MΩ-cm H_2O.

Procedure - 1) Weigh 19.3025g KCl into a beaker and pour the salt into a 1000 ml volumetric which already has ~500 ml H_2O. Rinse the remaining salt out of the beaker into the volumetric and fill the volumetric to 1000 ml.

$$19.3025\ g\ KCl * \frac{mol}{74.5513\ g} * \frac{1}{1\ L} * \frac{39{,}098.3\ ppm}{mol} = 10{,}123\ ppm$$

2.) Actually, I was shooting for ~.25 M KCl which 10,123 ppm is close too.

$$10{,}123 \frac{ppm}{L} * \frac{1\ mol}{39098.3\ ppm} = .258916 \frac{mol}{L}$$

cont.

3) Using the 10,123 ppm standard, dilute this to any concentration using various pipets and volumetrics.

4.) Instead of using the serial dilution method for making consecutively lower concentrations, ~~every concentration~~ T-6. each solution (or STD) T-6. was prepared directly from the 10,123 ppm STD.

5.)

$10{,}123 * \frac{50\ ml}{1000\ ml} = 1012.3\ ppm \doteq 1010\ ppm$ T-6.

$10{,}123 * \frac{50\ ml}{1000\ ml} = 506.15\ ppm \doteq 506\ ppm$

$10{,}123 * \frac{25\ ml}{1000\ ml} = 253.075\ ppm \doteq 253\ ppm$

$10{,}123 * \frac{10\ ml}{1000\ ml} = 101.23\ ppm \doteq 101\ ppm$

$10{,}123 * \frac{1\ ml}{1000\ ml} = 10.12\ ppm \doteq 10.1\ ppm$

$10{,}123 * \frac{300\ ml}{2000\ ml} = 1.518 \doteq 1.52\ ppm$

$10{,}123 * \frac{5\ ml}{1000\ ml} = 50.615 \doteq 50.6\ ppm$

6.)

8/14/91 1600 T.G.

K^+ Concentration from 80°C ISOTHERMAL EXP.

Equipment - Ion Selective Meter (S/N 001606)
Potassium Electrode

NaCl/KCl @ 80°C

E_{K^+}	Dilution Factor			Actual Diluted			Corrected Measurement			
	.5	.05	.005	0.5N	0.05N	0.005N	0.5N	0.05N	0.005N	
.01	1/10	1	1	22.2	20.8	2.33	222	20.8	2.33	
.02	1/10	1	1	47.0	40.2	4.36	470	40.2	4.36	TG 7-24-94
.03	1/10	1	1	86.5	67.3	7.39	865	~~67.3~~ 86.5	7.39	
.05	1/10	1	1	116	119	13.3	1160	~~1190~~ 1160	13.3	TG 7-24-94
.075	1/10	1	1	178	188	20.8	1780	188	20.8	
.09	1/10	1	1	219	222	23.8	2190	222	23.8	
.1	1/10	1	1	235	236	27.0	2350	236	27.0	
.13	1/10	1/10	1	281	27.9	32.3	2810	279	32.3	
.15	1/10	1/10	1	357	34.9	39.0	3570	347	39.0	
.18	1/10	1/10	1	475	38.7	43.9	4750	387	43.9	
.2	1/10	1/10	1	?450	44.3	50.0	4500	443	50.0	
.24	1/10	1/10	1	558	50.8	56.9	5580	508	56.9	
.3	1/10	1/10	1	685	?39.0	67.2	6850	390	67.2	
.35	1/10	1/10	1	825	64.5	75.5	8250	645	75.5	
.4	1/10	1/10	1	920	75.3	86.0	9200	753	86.0	
.5	.5/10	1/10	1	544	94.0	103	10880	940	103	
.6	.5/10	1/10	1	653	113	124	13060	1130	124	
.7	.5/10	1/10	1	722	135	143	14440	1350	143	
.75	.5/10	1/10	1	802	146	150	16040	1460	150	
.8	.5/10	1/10	1	906	153	161	18120	1530	161	
.9	.5/10	1/10	1	917	172	179	18340	1720	179	
				Avg slopes			: 57.3	: 56.9	: 58.2	

8/16/91 0800 T.G.

Follow the procedures from pg 112 & 113 ~~in order~~ taking the NaCl/$CaCl_2$ solutions from the 80°C oil bath, and make the dilutions for each solution accordingly. Label the bottles.
e.g.

$E_{Ca^{2+}}$: .015 .05N 80°C → $E_{Ca^{2+}}$: .015 .05N 80°C Na^+ & $E_{Ca^{2+}}$: .015 .05N 80°C Ca^{2+}

where Na^+ and Ca^{++} represent the respective ISA which will be added to the bottles for measurement.

8/17/91 0900 T.G.

Preparation of Na^+ Standards

Objective - to prepare various Na^+ standards used in calibrating the Ion Selective Meter.

Method - Dilute different volumes of 1000ppm NaCl standard.

Equipment - Volumetric flasks
Class A pipets (100, 50, 5 ml.)

Reagents - 1000 ppm Sodium Standard (Orion)
4 L of 17 MΩ-cm H_2O

cont.

Procedure – 1.) Using the bought sodium standard, dilute various volumes of the 1000 ppm in volumetric flasks.

2.) Instead of using the serial dilution method for making consecutively lower concentrations, each solution was prepared directly ~~from~~ T.G. 7-24-94 from the 1000 ppm Orion solution.

3.) The solutions made were:

$1000 * \frac{50ml}{100ml} = 500 ppm$

$1000 * \frac{100ml}{500ml}$ T.G. 7-24-94 $= 200 ppm$

$1000 * \frac{50ml}{1000ml} = 50 ppm$

$1000 * \frac{5ml}{1000ml} = 5 ppm$

A 100 ppm standard was not prepared since we also have a purchased 100 ppm standard from Orion.

8/17/91 1300 T.G.

Na^+ Concentration from NaCl/KCl, 80°C ISOTHERM EXPERIMENT

Equipment – Ion Selective Meter (S/N 001606)
Sodium Electrode

NaCl/KCl @ 80°C

Ext	Dilution Factor			Actual Diluted			Corrected Measurement		
	Na^+	Nat	Nat	.5N	.05N	.005N	.5N	.05N	.005N
.01	5/10	1/10	1	587	115	117	11740	1150	117
.02	5/10	1/10	1	613/618	114	117	12260/12360	1140	117
.03	5/10	1/10	1	709/702	114	113	14180/14040	1140	113
.05	5/10	1/10	1	567	109	110	11340	1090	110
.095	5/10	1/10	1	548	105	106	10960	1050	106
.098 T.G.	5/10	1/10	1	558/560	104	104	11160/11200	1040	104
.1	5/10	1/10	1	539	102	102	10780	1020	102
.13	5/10	1/10	1	522	98.8	97.5	10440	988	97.5
.15	5/10	1/10	1	530	98.6	93.9	10600	986	93.9
.18	5/10	1/10	1	578/573	93.4	91.0	11560/11460	934	91.0
.2	5/10	1/10	1	475	93.9	88.8	9500	939	88.8
.24	5/10	1/10	1	461	87.1	83.4	9220	871	83.4
.3	1/10	1/10	1	869	81.2	76.5	8690	812	76.5
.35	1/10	1/10	1	863	72.3	71.2	8630	723	71.2
.4	1/10	1/10	1	755	65.2	64.6	7550	652	64.6
.5	1/10	1/10	1	592	56.6	54.6	5920	566	54.6
.6	1/10	1/10	1	462	44.0	44.2	4620	440	44.2
.7	1/10	1/10	1	833	32.4	33.4	3330	324	33.4
.75	1/10	1/10	1	290	27.0	27.8	2900	270	27.8
.8	1/10	1/10 T.G.	1	248	219	22.0	2480	219	22.0
.9	1/10	1/10 T.G.	1	111	114	11.2	1110	114	11.2

Avg. Slopes: 59.0 : 58.8 : 58.7

8/28/91 0800 T.G.

Preparation of Ca^{2+} Standards

Objective - to prepare various Ca^{2+} standards used in calibrating the Ion Selective Meter. TG 7-24-94

Method - Dilute ~~4010 ppm~~ TG 7-24-94 .1M Calcium Chloride to different concentration.

Equipment - Volumetric flasks (Class A)
Class A Pipets.

Reagents - .1M $CaCl_2$ Standard (Orion)
8L of 17MΩ-cm H_2O.

Procedure - 1) Using the bought Calcium Chloride standard, dilute various volumes of the 4010 ppm solution in volumetric flasks.

2) The solutions made were:

4010 * 50/200 = 1002.5 ≅ 1000 ppm
4010 * 50/250 = 802 ≅ 802 ppm
4010 * 50/500 = 401 = 401 ppm
4010 * 50/1000 = 200.5 ≅ 201 ppm
4010 * 25/1000 = 100.25 ≅ 100 ppm
4010 * 10/1000 = 40.1 = 40.1 ppm
4010 * 1/1000 = 4.01 = 4.01 ppm
4010 * 100 µl/1000 ml = .401 = .401 ppm.

8/29/91 1200 T.G.

Na^+ Concentration from $NaCl/CaCl_2$, 80°C
ISOTHERM EXPERIMENT

Equipment - Ion Selective Meter (S/N 001606)
Sodium Electrode

$NaCl/CaCl_2$ @ 80°C

$E_{Ca^{2+}}$	$E_{Ca^{2+}}$	$E_{Ca^{2+}}$	Dilution Factor			Actual Diluted			Corrected Actual		
.5	.05	.005	.5	.05	.005	.5	.05	.005	.5	.05	.005
.02	.015	.01	.5/10	1/10	1	605	113	118	12100	1130	118
.04	.03	.03	.5/10	1/10	1	579	113	116	11580	1130	116
.06	.05	.05	.5/10	1/10	1	577	111	115	11540	1110	115
.08	.08	.08	.5/10	1/10	1	573	108	113	11460	1080	113
.1	.1	.1	.5/10	1/10	1	549	108	111	10980	1080	111
.12	.15	.13	.5/10	1/10	1	552	105	103	11040 TG 7-24-94	1050	103
.15	.2	.17	.5/10	1/10	1	510	98.1	104	10200	981	104
.2	.25	.2	.5/10	1/10	1	573	90.5	99.8	11460	905	99.8
.25	.3	.23	.5/10	1/10	1	476	85.7	100	9520	857	100
.3	.4	.27	.5/10	1/10	1	451	75.3	94.0	9020	753	94.0
.4	.5	.3	.5/10	1/10	1	–	63.2	92.3	–	632	92.3
.5	.55	.35	.5/10	1/10	1	–	56.9	85.7	–	569	85.7
.6	.6	.4	.5/10	1/10	1	539	51.8	83.0	10780	518	83.0
.65	.65	.45	.5/10	1/10	1	459	47.9	73.0	9180	479	73.0
.7	.7	.5	.5/10	1/10	1	–	40.5	67.8	–	405	67.8
.75	.75	.6	.5/10	1/10	1	169	33.2	53.3	9380	332	53.3
.8	.8	.65	.5/10	1/10	1	147	25.6	45.8	2940	256	45.8
.83	.85	.7	.5/10	~~1/10~~ 1 TG 7-24-94	1	–	186	38.8	–	186	38.8
.87	.9	.75	1/10	~~1/10~~ 1 TG 7-24-94	1	181	117	31.5	7-24-94 TG 7.6 1810 ~~3620~~	117	31.5
.9	.95	.8	1/10	1	1	134	58.9	25.1	1340	58.9	25.1
.93		.85	1/10	≠	1	88.7		18.2	887		18.2
.95		.9	1/10	≠	1	60.3		11.6	603		11.6
		.95			1			5.79			5.79

TG 7-24-94

8/21/91 1600 T.G.

Ca^{2+} Concentration from $NaCl/CaCl_2$, 80°C

ISOTHERM EXPERIMENT

Equipment - Ion Selective Meter (S/N 001606)
Calcium Electrode

$NaCl/CaCl_2$ @ 80°C

$E_{Ca^{2+}}$			Dilution Factor			Actual Diluted			Corrected Actual		
.5	.05	.005	.5	.05	.005	.5	.05	.005	.5	.05	.005
.02	.015	.01	10.9	1	1	.5/10	7.57	.112	109	7.57	.112
.04	.03	.03	23.1	1	1	.5/10	15.9	.376	231	15.9	.376
.06	.05	.05	34.1	1	1	.5/10	28.1	.656	341	28.1	.656
.08	.08	.08	56.5	1	1	.5/10	45.3	1.75	565	45.3	1.75
.1	.1	.1	68.7	1	1	.5/10	64.6	3.75	687	64.6	3.75
.12	.15	.13	88.7	1	1	.5/10	107	4.79	887	107	4.79
.15	.2	.17	117	1	1	.5/10	136	9.24	1170	136	9.24
.2	.25	.2	177	1	1	.5/10	192	12.6	1770	192	12.6
.25	.3	.23	204	1	1	.5/10	229	11.5	2040	229	11.5
.3	.4	.27	237	1/10	1	.5/10	31.3	16.3	2370	313	16.3
.4	.5	.3	—	1/10	1	.5/10	41.3	17.9	—	413	17.9
.5	.55	.35	TG 7-24-94	1/10	1	.5/10	47.1	23.5	—	471	23.5
.6	.6	.4	271 T.G.	1/10	1	.5/10	53.0	26.6	2710	530	26.6
.65	.65	.45	~~734~~ (307 TG)	1/10	1	.5/10	55.9	33.7	3070	559	33.7
.7	.7	.5	—	1/10	1	.5/10	61.1	37.7	—	611	37.7
.75	.75	.6	734	1/10	1	.5/10	66.7	50.9	7340	667	50.9
.8	.8	.65	779	1/10	1	.5/10	73.0	56.6	7790	730	56.6
TG 7-24-94 .85	.85	.7	—	1/10	1	.5/10	81.4	63.1	—	814	63.1
.87	.9	.75	408	1/10	1	.5/10	85.1	69.4	8160	851	69.4
TG 7-24-94 .95	.95	.8	444	1/10	1	.5/10	89.0	74.8	8880	890	74.8
.93		.85	463		1	.5/10		80.4	9260		80.4
.95		.9	475		1	.5/10		86.3	9500		86.3
		.95			1	TG 7-24-94		90.9			90.9

Switch TG. 8/21/91

8/22/91 0800 T.G.

After viewing the constructed isotherms from the 80°C measurements, it was decided that some ~~should be~~ T.G. solutions T.G. should be reanalyzed. Therefore, some selected solutions were prepared and placed in a bath @ 80°C.

8/22/91 1630, T.G.

Preparation of Various CDV--$NaCl/CaCl_2$ (0.5) Mixtures for Isotherm Experiment @ 80°C

$NaCl/CaCl_2$ (0.5N @ 80°C) ISOTHERM EXP.

$E_{Ca^{2+}}$	$E_{i,Ca^{2+}}$	wt. zeol. to use (g)	vol. sol'n used (ml)	wt zeol used (g)	wt. sol'n used (g)
.12	.3	1.037	5	1.0372	5.0983
.2	.4	0.9601	5	.9601	5.0868
.4	.6	0.6878	5	.6877	5.0898
.5	.7	0.5985	5	.5985	5.0843
.6	.8	0.5238	5	.5238	5.0876
.65	.8	0.3677	5	.3677	5.0883
.7	.9	0.4655	5	.4655	5.0809
.83	1.0	0.3377	5	.3377	5.0854
.87	1.0	0.4911	10	.4911	10.1783

cont. Preparation of the CDV-- NaCl/KCl (0.5N) Mixtures for ISOTHERM EXPERIMENT @ 80°C

Everything, including the temperature, is the same as that found on pg 100.

NaCl/KCl (0.5N @ 80°C) ISOTHERM EXPERIMENT

E_{k+} T.G.	$E_{i,k+}$	wt. zeol to use (g)	Vol sol'n used (ml)	wt. zeol. used (g)	wt. sol'n used (g)
.01	.1	1.037	10	1.0373	10.1217
.02	.1	0.6144	10	.6145	10.1290
.03	.1	0.4032	10	.4031	10.1222
.05	.1	0.2259	10	.2258	10.1314
.075	.2	0.4800	10	.4799	10.1581
.09	.2	0.3960	10	.3960	10.1721
.1	.2	0.3518	10	.3519	10.1728
.13	.2	0.2304	10	.2304	10.1595
.15	.3	0.4735	10	.4734	10.1680
.18	.3	0.3687	10	.3687	10.1690
.2	.3	0.2992	10	.2992	10.1821
.24	.4	0.4608	10	.4607	10.1665
.3	.4	0.2793	10	.2793	10.1846
.35	.5	0.4066	10	.4066	10.1756
.4	.6	0.5297	10	.5297	10.1691
.5	.7	0.5149	10	.5149	10.1795
.6	.9	0.7514	10	.7513	10.1926
.7	1.0	0.7354	10	.7355	10.1820
.75	1.0	0.6064	10	.6066	10.1870
.8	1.0	0.4800	10	.4801	10.1931
.9	1.0	0.2351	10	.2351	10.1845

cont. Preparation of Various CDV-- NaCl/KCl (0.005N) Mixtures for ISOTHERM EXPERIMENT @ 80°C

NaCl/KCl (0.005N @ 80°C) ISOTHERM EXP.

E_{k+}	$E_{i,k+}$	wt. zeol. to use (g)	vol. sol'n ~~wt. zeol~~ used (ml) T67-24-94	wt. zeol used (g)	wt. sol'n used (g)
.6	1.0	.1002	100	.1002	99.6163
.7	1.0	.0735	100	.0734	99.6216
.75	1.0	.0606	100	.0607	99.6131
.8	1.0	.1200	250	.1198	248.63
.9	1.0	.0588	250	.0590	248.57

8/25/91 1700 T.G.

The solutions were taken out of the 80°C oil bath in the same way that was discussed on pgs 112 and 113. Dilution was performed on those solutions that required it. Follow the procedures given on the previously mentioned pages.

The concentrations of the selected solutions follow.

8/27/91 1100 T.G.

Na⁺ Concentrations from the Various .5N NaCl/$CaCl_2$, .5N NaCl/KCl, and .005N NaCl/KCl

80°C ISOTHERM EXPERIMENT

Equipment - Same as pg 125.

Various NaCl Mixtures @ 80°C

$E_{Ca^{2+}}$	E_{K^+}	E_{K^+}	Dilution Factor			Actual Diluted			Corrected Actual		
.5	.5	.005	.5	.5	.005	.5 Na/Ca	.5 Na/K	.005 Na/K	.5 Na/Ca	.5 Na/K	.005 Na/K
.12	.01	.6	1/10	1/10	1	540	590	42.0	10800	11770	42.0
.2	.02	.7	1/10	1/10	1	505	575	31.5	10100	11500	31.5
.4	.03	.75	1/10	1/10	1	394	575	26.3	7880	11500	26.3
.5	.05	.8	1/10	1/10	1	333	564 T6	20.6	6660	11340	20.6
.6	.075	.9	1/10	1/10	1	—	544 T6	10.6	—	10880	10.6
.65	.09		1/10	1/10	*	—	539		—	10780	
.7	.1		1/10	1/10		216	T6: 539 546		4320	10780	
.83	.13		1/10	1/10		122	T6: 515 508		2440	10300	
.87	.15		1/10	1/10		187	488		1870	9760	
	.18			1/10			485			9760	
	.2			1/10			475			9500	
	.24			1/10			450			9000	
	.3			1/10			814			8140	
	.35			1/10			780			7800	
	.4			1/10			679			6790	
	.5			1/10			576.5			5765	
	.6			1/10			446			4460	
	.7			1/10			338			3380	
	.75			1/10			216			2760	
	.8			1/10			169			2160	
	.9			1/10						1090	

T6 7-24-94

Cont.

Ca^{2+} and K^+ from 7-24-94 concentrations from the .5N NaCl/$CaCl_2$, .5N NaCl/KCl, and .005N NaCl/KCl,

60°C ISOTHERM EXPERIMENT

Equipment - Same as pgs 120 & 126.

$E_{Ca^{2+}}$	E_{K^+}	E_{K^+}	Dilution Factor			Actual Diluted			Corrected 7-24-94 Actual		
.5	.5	.005	.5	.5	.005	.5 Ca^{2+}	.5 K^+	.005 K^+	.5 Ca^{2+}	.5 K^+	.005 K^+
.12	.01	.6	1/10	1/10	1	94.2	20.9	119	1884	209	119
.2	.02	.7	1/10	1/10	1	163	39.4	134	1630	394	134
.4	.03	.75	1/10	1/10	1	185	69.5	145	3700	675	145
.5	.05	.8	1/10	1/10	1	232	116	154	4640	1160	154
.6	.075	.9	1/10	1/10	1	—	179	168	—	1790	168
.65	.09		1/10	1/10		—	213		—	2130	
.7	.1		1/10	1/10		359	228		7180	2280	
.83	.13		1/10	1/10		401	269		8020	2690	
.87	.15		1/10	1/10		435	332		8700	3320	
	.18			1/10			383			3830	
	.2			1/10			466			4660	
	.24			1/10			515			5150	
	.3			1/10			622			6220	
	.35			1/10			751			7510	
	.4			1/10			829			8290	
	.5			1/10			500			10000	
	.6			1/10			616			12320	
	.7			1/10			742			14840	
	.75			1/10			764			15280	
	.8			1/10			794			15880	
	.9			1/10			880			17600	

T6 7-24-94

8/27/91 1700 T.G.

To ensure that my Potassium standards were correct, I diluted some .1M (3910 ppm) Corning Potassium standard and predicted what the ~~values~~ concentrations T.G. should be from those dilutions. I then had Jim Prikryl measure these solutions using the KCl standards that I prepared on 8/15/91.

The results were:

theoretical	experimental
391 ppm	387
782 ppm	788

Avg slope: 57.4 mV.

8/28/91 0900 JP

Recovery of clinoptilolite used in 80°C Ion Exchange Experiments

Followed procedures as outlined on p106 for recovery of clinoptilolite used in 90°C Ion Exchange experiments.

The recovered clinoptilolite was placed in the container containing CDV*100/200*UC*WA*HL*CPT*NaF*REC.

New weight of CDV*...*REC + container = 208.97 g

8/30/91 1550 hrs JP

Preparation of Calcium (CaF) and Potassium (KaF) Clinoptilolite

Objective: Replace all free cations in Death Valley clinoptilolite (CDV*100/200*UC*WA*HL*CPT) with Ca^{++} or K^{+} ions for ion exchange experiments.

Method: Saturate the clinoptilolite in 3M $CaCl_2 \cdot H_2O$ or 3M KCl at 90°C.

Equipment:
1) Versa Bath Model 236 constant temperature bath
2) 500 ml + 1L polypropyline bottles
3) Mettler PM 4600 balance accurate to .1g (SN-20467)

Reagents:
a) 3M $CaCl_2 \cdot H_2O$ - dissolve 882.12 g $CaCl_2 \cdot H_2O$ in 2L water. Store in 1L plastic bottles
b) 3M KCl - dissolve 447.36 g KCl in 2L water. Store in 1L plastic bottles.

Procedure:

1) Place 25.8365g CDV#...#CPT in a 500 ml plastic bottle and add about 400 ml of the 3M $CaCl_2 \cdot H_2O$ solution. Place 25.3911g CDV#...#CPT in a 500 ml plastic bottle and add about 400 ml of the 3M KCl solution.

2) Place these two bottles in the water bath at 90°C and 50 rpm.

3) Every 3 or 4 days for about 3 weeks replace the solution in the bottles by decanting the old solution and adding about 400 ml of 3M $CaCl_2 \cdot H_2O$ or 3M KCl to the respective bottle.

9/11/91 1600 hrs gp.

Determination of Chloride in 0.5N $CaCl_2$-NaCl reference solutions by gravimetric analysis.

Objective- To measure the chloride concentration of the 0.5N $CaCl_2$-NaCl solution used in ion-exchange experiments.

Method- Determine chloride gravimetrically by precipitating as silver chloride.

Equipment-

a) Mettler AE240 balance accurate to 0.1 mg (SN-101237)
b) Corning hot plate
c) Glass beakers, stir rod, erlenmeyer flask, pipettes
d) Aluminum foil
e) Glass fitten crucibles Type F 30 ml, rubber rings, suction flasks.
f) Blue M drying oven

Reagents-

a) 0.1M $AgNO_3$ Lot 910644-24
b) 1:1 HNO_3 Lot 910692 Add 1 part HNO_3 To 1 part H_2O
c) 2M HNO_3 Lot 910692 Dilute 15.8N HNO_3 to 2N

Procedure -

a) Pipette 2 ml of $NaCl-CaCl_2$ solution into a 600 ml beaker, add about 70-80 ml H_2O and heat nearly to boiling.

b) In a 250 ml beaker heat nearly to boiling approximately 125 ml of 0.1 M $AgNO_3$ solution acidified with 1 ml dilute (1:1) HNO_3.

c) Pour the $AgNO_3$ solution down a glass rod into the $NaCl-CaCl_2$ solution and stir center of beaker vigorously for several minutes without brushing the sides.

d) Coagulation of large curdy flakes of AgCl form and settle during heating and stirring. When coagulation occurs stop heating & test for completeness of precipitation by adding a few drops of $AgNO_3$ solution. If turbidity occurs, add a few more ml of $AgNO_3$, stir the solution, and test for completness of precipitation, etc..

e) When precipitate is complete cover the beaker with aluminum foil and store overnight in a dark area to protect from light.

f) Wash filter crucibles thoroughly with back suction, finish with hot dilute HNO_3 (5 ml of 2N solution to 100 ml H_2O) and then with hot water, and dry at 130°C to constant weight. Record weight

g) Place filter crucible in neck of flask & filtrate. Decant liquid. Wash precipitate several times by decantation with hot dilute HNO_3. Transfer precipitate to filter crucible and wash with dilute HNO_3 and then with H_2O.

h) Dry crucible at 130°C to constant weight and find weight of AgCl from the difference between weights of empty crucible and crucible with AgCl. Record weight

i) Determine amount of chloride using the following formula:

$$\text{Weight of Chloride} = \text{Weight of AgCl} \times \frac{35.453}{143.321}$$

Results -

$X_{Cu} = 0.4$

weight of crucible = 31.2850
weight of crucible + AgCl = 31.4278
weight of AgCl = 0.1428

weight of chloride = $0.1428 \times \frac{35.453}{143.321} = .035324$

$X_{Cu} = 0.5$

weight of crucible = 31.0557
weight of crucible + AgCl = 31.1989
weight of AgCl = 0.1432

weight of chloride = $0.1432 \times \frac{35.453}{143.321} = .035423$

$X_{Cu} = 0.6$

weight of crucible = 31.0906
weight of crucible + AgCl = 31.2337
weight of AgCl = 0.1431

weight of chloride = $0.1431 \times \frac{35.453}{143.321} = .035398$

$X_{Cu} = 0.7$

weight of crucible = 31.0553
weight of crucible + AgCl = 31.1990
weight of AgCl = 0.1437

weight of chloride = $0.1437 \times \frac{35.453}{143.321} = .035547$

$X_{Cu} = 0.8$

weight of crucible = 31.0075
weight of crucible + AgCl = 31.1502
weight of AgCl = 0.1427

weight of chloride = $0.1427 \times \frac{35.453}{143.321} = 0.035299$

$X_{Cu} = 0.9$

weight of crucible = 31.0558
weight of crucible + AgCl = 31.1998
weight of AgCl = 0.1440

weight of chloride = $0.1440 \times \frac{35.453}{143.321} = 0.035621$

$X_{Cu} = 1.0$

weight of crucible = 31.2846
weight of crucible + AgCl = 31.4286
weight of AgCl = 0.1440

weight of chloride = $0.1440 \times \frac{35.453}{143.321} = 0.035621$

$X_{Cu} = 0.1$

weight of crucible = 31.0549
weight of crucible + AgCl = 31.1997
weight of AgCl = 0.1448

weight of chloride = $0.1448 \times \frac{35.453}{143.321} = 0.035819$

$X_{Ca} = 0.2$

weight of crucible = 31.0896
weight of crucible + AgCl = 31.2304
weight of AgCl = 0.1408

weight of chloride = $0.1408 \times \frac{35.453}{143.321} = 0.034829$

$X_{Ca} = 0.3$

weight of crucible = 30.9846
weight of crucible + AgCl = 31.1273
weight of AgCl = 0.1427

weight of chloride = $0.1427 \times \frac{35.453}{143.321} = 0.035299$

9/[illegible]/91 0950 hrs JP

Decanted solutions (3M KCl + 3M $CaCl_2 \cdot H_2O$) from the polypropylene bottles containing CDV* ... * CPT (see pp. 133-134) and refilled bottles with about 400 ml H_2O. Placed the bottles back into the water bath at 90°C

9/13/91 1330 hrs JP

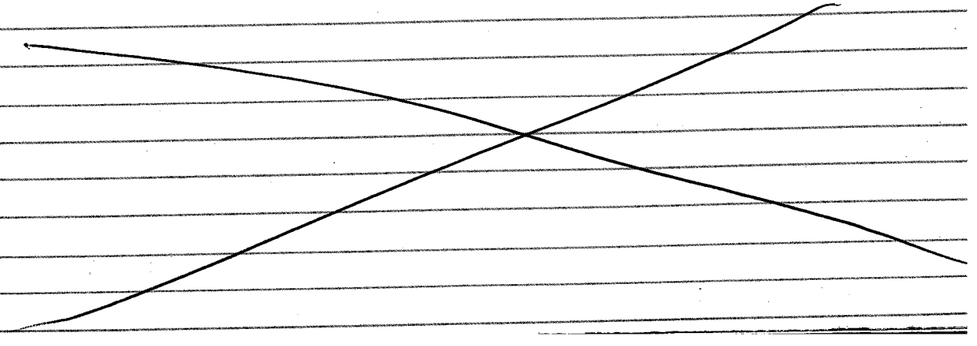
Took 3M KCl + 3M solutions containing Ca + K form clinoptilolite out the 90°C bath and washed the clinoptilolite. Washing was performed by decanting each bottle and replacing with ~400 ml H_2O. The bottle was shaken to mix and bring the clinoptilolite into suspension. The clinoptilolite was then allowed to settle This procedure was repeated 6 times.

9/13/91 1400 hrs JP

After the clinoptilolite in each bottle was washed 6 times the solutions were checked for chloride content. About 5 to 10 ml of solution was decanted into a small beaker and 0.1 N $AgNO_3$ was added dropwise to test for AgCl precipitation. If a white AgCl precipitate forms on addition of $AgNO_3$ then the solution contains chloride. The $AgNO_3$ test was negative for both solutions. The solutions were again placed in the 90°C bath.

9/13/91 1630 hrs JP

Recheck solutions containing Ca-K + K-F clinoptilolite. Decant 5 to 10 ml of solutions into beaker and add 0.1 N $AgNO_3$ dropwise to test for AgCl precipitate. The $AgNO_3$ test was negative for both solutions. Placed bottles back into 90°C bath and allow to set over weekend.



9/16/91 0900hrs JP

Took out CaF + KF clinoptilolite solutions to check for chloride. Decanted 5-10 ml of each solution + added a few drops of 0.1 N $AgNO_3$ to test for AgCl precipitation. CaF solution became cloudy upon addition of $AgNO_3$. The solutions in each bottle were changed and placed back into the 90°C bath.

9/16/91 1330hrs JP

Took out CaF + KF clinoptilolite ~~solutions~~ to check for chloride using $AgNO_3$ ~~test~~ Test was negative for both solutions.

9/16/91 1400hrs JP

Took CaF + KF clinoptilolite solutions, swirled each to bring clinoptilolite into suspension and ultrasonically cleaned for 5 minutes. Decanted each solution and replaced with H_2O. Repeated ultrasonic cleaning 5 times. Washed with H_2O and transferred to 250 ml. beakers. Let suspension settle, decanted liquid + dried in oven overnight at ~60°C.

9/17/91 0900hrs JP

CaF + KF clinoptilolite samples were taken out of oven. Beakers were covered with parafilm and labeled CDV*100/200*UC*WA*HL*CPT*KF and CDV*100/200*UC*WA*HL*CPT*CaF

Samples were allowed to cool and weighed

CDV*...*KF = 89.1472 g

CDV*...*CaF = 79.2962 g

Samples were left open and allowed to equilibrate with H_2O over saturated NaCl by placing in a dessicator containing saturated NaCl

Samples will be weighed periodically to determine equilibrium with H_2O

		CDV*...*KF	CDV*...*CaF
1403 hrs	9/17/91	89.3683 g	79.5239 g
1507 hrs	9/18/91	90.0089 g	80.1268 g
0908 hrs	9/19/91	90.1452 g	80.2488 g
0800 hrs	9/20/91	90.2067 g	80.3130 g
0800 hrs	9/23/91	90.2512 g	80.3519 g
0800 hrs	9/24/91	90.2529 g	80.3529 g
0830 hrs	9/25/91	90.2546 g	80.3547 g
0800 hrs	9/26/91	90.2481 g	80.3508 g
0830 hrs	9/27/91	90.2453 g	80.3481 g
0805 hrs	10/1/91	90.2666 g	80.3663 g

11/20/91
0810 hrs JP

Preparation of $NaNO_3$-KNO_3 and $NaNO_3$-$CaNO_3$ Solutions.

Objective: Prepare solutions to be used in isotherm experiments

Equipment:
a) Mettler AE240 balance accurate to 0.1 mg (SN-101237)

Materials + Supplies:
a) Sodium Nitrate ($NaNO_3$) - lot NO 7808 KDJE
b) Potassium Nitrate (KNO_3) - lot NO 7028 KDPD
c) Calcium Nitrate ($CaNO_3$) - lot NO 42316 ICEK
d) 250 ml beakers
e) stir bars and Corning stirrer
f) 250 ml volumetric flasks
g) glass rods
h) 250 ml polypropylene (PP) bottles
i) 1000 ml PP bottles
j) 500 ml PP bottles
k) glass pipets

Procedure:
1) Prepared 250 ml of $NaNO_3$-KNO_3 and $NaNO_3$-$CaNO_3$ aqueous mixtures with a total normality of 0.5N and a fixed Na/K or Na/Ca ratio by toring reagent grade $NaNO_3$, KNO_3, and $CaNO_3 \cdot 4H_2O$ in amounts given in Tables 1 and 2 onto a acid washed 250 ml beaker. Beaker were kept covered with parafilm between additions of reagents to minimize adsorption of water from the atmosphere.
Added about 100 ml of ultrapure water to each beaker, added a stir bar and dissolved salts completely on a stir plate. Retrieved the stir bar in each beaker with a magnetic rod and carefully rinsed the rod + stir bar with ultrapure water making sure to catch all washes in the beaker.

Decanted solutions into clean 250 ml volumetric flasks with the help of a glass rod; rinsed beakers + glass rods with ultrapure water several times, carefully transferring all washes to the volumetric flasks. Filled flasks to about 2-3 inches below mark with ultrapure water; swirled all about flasks. Let flasks stand for a few seconds and then added water dropwise to the mark; remixed.

Table 1

DATE PREPARED

X_K (0.5 N) ($NaNO_3$-KNO_3 soln.)	Wt. $NaNO_3$ needed for 250 ml	Wt. KNO_3 needed for 250 ml	Wt. $NaNO_3$ used	Wt. KNO_3 used
0.01	10.518	0.12638	~~10.5183~~ ~~10.5184~~ 11/20/91 TD	.1263
0.025	10.359	0.31594	10.3591	.3156
0.05	10.093	0.63189	10.0931	.6317
0.075	9.8274	0.94783	9.8277	.9478
0.10	9.5618	1.2638	9.5620	1.2639
0.20	8.4994	2.5276	8.4997	2.5274
0.30	7.437	3.7913	7.4368	3.7912
0.40	6.3746	5.0551	6.3747	5.0553
0.50	5.3121	6.3189	5.3124	6.3187
0.60	4.2497	7.5827	~~4.2498~~ TD 11/21/91 4.2499	7.5830
0.70	3.1873	8.8464	3.1873	8.8463
0.80	2.1248	10.11	~~2.1251~~ TD 11/21/91 2.1249	10.1104
0.90	1.0624	11.374	1.0621	11.3740
0.925	0.79682	11.69	.7967	11.6903
0.95	0.53121	12.006	.5314	12.0064
0.975	0.26561	12.322	.2658	12.3224
1.00	0	12.638	0	12.6381

Table 2

DATE PREPARED

X_{Ca} (0.5 N) ($NaNO_3$-$Ca(NO_3)_2$ soln.)	Wt. $NaNO_3$ needed for 250 ml	Wt. $Ca(NO_3)_2 4H_2O$ needed for 250 ml	Wt. $NaNO_3$ used	Wt. $Ca(NO_3)_2 4H_2O$ used
0.01	10.518	0.14759	~~10.5184~~ TD 11/25/91 10.5185	~~.1475~~ TD 11/25/91 .1478
0.025	10.359	0.36898	10.3589	.3693
0.05	10.093	0.73796	10.0934	.7382
0.075	9.8274	1.1069	9.8276	1.1072
0.1	9.5618	1.4759	9.5619	~~1.4758~~ TD 11/25/91 1.4760
0.2	8.4994	2.9519	8.4998	2.9518
0.3	7.437	4.4278	7.4372	4.4279
0.4	6.3746	5.9037	6.3749	5.9039
0.5	5.3121	7.3796	5.3123	7.3796
0.6	4.2497	8.8555	4.2496	8.8558
0.7	3.1873	10.331	3.1873	10.3309
0.8	2.1248	11.807	2.1252	11.8073
0.9	1.0624	13.283	1.0624	13.2833
0.925	0.79682	13.652	.7967	13.6517
0.95	0.53121	14.021	.5315	14.0213
0.975	0.26561	14.39	.2654	14.3901
1	0	14.759	0	14.7589

11/22/91 0930hrs

2) 1000ml of .05N ~~Na-NO3~~ TD 11/22/91 $NaNO_3-KNO_3$ (or $NaNO_3-Ca(NO_3)_2$) solution were made by pipetting 100 ml of the .5N solution and diluting it with ultrapure water in a 1000ml volumetric flask (The pipette was rinsed with a few ml of the .5N solution). The .05N solution was transferred to a 1000ml pp bottle and labeled (eg. $NaNO_3/KNO_3$ * .05N * .1X_K plus date and initial)

11/25/91 TJD

Preparation of Zeolite Samples to be sent to Texas Tech Univ for DCP analysis

Method: approximately 0.5 gm of the zeolite material is placed in a screw-cap glass vial. The vials are labeled A to J. This page of the lab notebook (GC-04-148) is included in the label for reference. Two of the vials have NBS standards. The vial labels and the corresponding sample identification are given below:

A – CDV*100/200*UC*WA*HL*CPT*Naf
B – K-feldspar; NBS 70A
C – CDV*100/200*UC*WA*HL*CPT*H1
D – CDV*100/200*UC*WA*HL*CPT*Hf
E – CDV*100/200*UC*WA*HL*CPT*Naf
F – CDV*100/200*UC*WA*HL*CPT*Hf
G – CDV*100/200*UC*WA*HL*CPT*H2
H – CDV*100/200*UC*WA*HL*CPT*Naf
I – CDV*100/200*UC*WA*HL*CPT*Hf
J – Na-feldspar; NBS 99A

Not done TJD 11/25/91

11/27/91 1000hrs JP

Preparation of CDV*...*Naf – $NaNO_3-KNO_3$ (0.05N) samples @ 25°C.

Objective – prepare samples consisting of CDV*...*Naf powder in 0.05N $NaNO_3-KNO_3$ solutions for isotherm experiments at 25°C.

Method – add CDV*...*Naf powder to 0.05N NO_3 solutions containing varying concentrations of Na and K and allow the sample to equilibrate at 25°C. JP 11/27/91

Equipment –
a) Mettler AE240 balance accurate to 0.001g (SN 101237)
b) 30 ml PP bottles
c) 25 ml pipets (class A)
d) spatulas
e) Versa Bath S, Model 236 (constant temperature bath)

Reagents and Supplies
a) 0.1 – 1.0 X_K 0.05N $NaNO_3-KNO_3$ solutions prepared previously (p. 148).
b) CDV*...*Naf powder

Procedure -

1) Acid clean and dry 30 ml PP bottle and 25 ml pipets

2) Label PP bottle as shown in Table 1 (eg $KNaNO_3$ *0.05*1)
For each sample

3) Weigh bottle empty with cap and record in Table 1

4) Remove cap from bottle, tare balance, and add CDV*...*Na+ weight shown in Table 1. Record CDV*N.*Na+ weight in Table 1.

5) Remove bottle, add Eix solution volume shown in Table 1 to bottle. Place cap on bottle and weigh and record weight in Table 1.

6) Place bottles in a constant Temperature bath set at 25°C and 60 rpm

Table 1

$NaNO_3$-KNO_3 (0.05 N) Isotherm Experiment

Sample Label $KNaNO_3$* 0.05*	E_{ix} used	Wt. Zeol. to use (gm)	Vol. Soln. Used (ml)	Wt. Zeol. Used (gm)	Wt. Bottle Empty (gm)	Wt. Bottle + Soln. (gm)	Sample Dilution Factor		Diluted Sample Concentration (ppm)		Reference Dilution Factor		Diluted Reference Concentration (ppm)	
							Na+	K+	Na+	K+	Na+	K+	Na+	K+
1	.1	.2592	25	.2595 11/27/01 .2590 TD	7.9586 11/27/01 8.0549 TD	33.1465 11/27/01 33.2579 TD	1/10	1	123	116	10/100	1	97.6	171
2	.1	.1536	25	.1537 11/27/01 .1536 TD	8.0297 11/27/01 7.9982 TD	33.1285 11/27/01 33.0863 TD	1/10	1	123	203	-	-	-	-
3	.1	.1008	25	.1012 11/27/01 .1010 TD	7.9935 11/27/01 7.9917 TD	33.0697 11/27/01 32.9913 TD	1/10	1	123	242	-	-	-	-
4	.2	.1694	25	.1695 11/27/01 .1696 TD	8.0416 11/27/01 7.9772 TD	33.0211 11/27/01 33.0525 TD	1/10	1	121	279	1	1	909	352
5	.3	.2160	25	.2159	8.0082	33.1245	1/10	1	119	318	1	1	798	533
6	.3	.1890	25	.1892	8.0330	32.9697	1/10	1	117	337	-	-	-	-
7	.3	.1759	25	.1762	7.9875	33.1282	1/10	1	115	345	-	-	-	-
8	.3	.1399	25	.1397	7.9552	33.0141	1/10	1	113	372	-	-	-	-
9	.4	.1972	25	.1974	7.9909	32.9025	1/10	1	108	380	1	1	663	699
10	.4	.1690	25	.1691	8.0303	33.1077	1/10	1	106	503	-	-	-	-
11	.4	.1496	25	.1498	8.0313	33.0493	1/10	1	102	525	-	-	-	-
12	.4	.1152	25	.1153	7.9351	32.8564	1/10	1	97.8	633	-	-	-	-
13	.5	.1397	25	.1397	7.8976	32.9582	1/10	1	88.7	786	1	1	545	875
14	.6	.1694	25	.1695	7.9720	33.1710	1/10	1	80.8	908	1	10/100	436	98.2
15	.7	.1897	25	.1899	7.9672	33.1047	1/10	1	69.4	1050	1	10/100	328	115
16	.8	.1931	25	.1929	8.0230	33.0274	1/10	1/10	55.8	98.1	1	10/100	218	128
17	.9	.1879	25	.1877	7.9722	32.9705	1/10	1/10	44.3	117	1	10/100	108	143
18	1.0	.1838	25	.1838	8.0217	33.1981	1/10	1/10	32.3	135	-	10/100	-	162
19	1.0	.1516	25	.1519	8.0182	33.0887	1/10	1/10	26.6	143	-	-	-	-
20	1.0	.1200	25	.1201	7.9736	32.9551	1/10	1/10	20.9	155	-	-	-	-
21	1.0	.0588	25	.0589	8.0301	32.9869	1/10	1/10	10.4	175	-	-	-	-

11/27/91 1300hrs TD

Preparation of CDV*...*Naf - $NaNO_3$-$Ca(NO_3)_2$ (0.05 N) Samples at 25°C.

TD 11/27/91

Objective - prepare samples ~~of~~ consisting of CDV*...*Naf powder in 0.05 N $NaNO_3$-$Ca(NO_3)_2$ solutions for isotherm experiments at 25°C.

Method - add CDV*...*Naf powder to 0.05 N NO_3 solutions containing varying concentrations of Na and Ca and allow the sample to equilibrate at 25°C

Equipment

a) Mettler AE240 balance accurate to 0.001g (SN 101237)
b) 30 ml PP bottles
c) 60 ml PP bottles
d) 25 ml pipets (class A)
e) 50 ml pipette
f) spatulas
g) Versa Bath S, Model 236 (constant temperature bath)

Reagents and Supplies

a) 0.1 - 1.0 X_{Ca} 0.05N $NaNO_3$-$Ca(NO_3)_2$ solutions prepared previously (p. 148)

b) CDV*...*Naf powder

Procedure -

1) Acid clean and dry 30ml PP bottles, 60ml PP bottles, 25ml pipettes and 50ml ~~pipp~~ pipettes TD 11/27/91

2) Label PP bottles as shown in Table 1 TD
(eg $CaNaNO_3$ * 0.05 * 1)

3) For each sample, weigh bottle empty with cap and record in Table 1

4) Remove cap from bottle, tare balance, and add CDV*...*Naf weight shown in table 1. Record CDV*...*Naf weight in table 1.

5) Remove bottle, add $E_{i,Ca}$ solution volume shown in Table 1 to bottle. Place cap on bottle, weigh and record weight in Table 1.

6) Place bottles in a constant temperature bath set at 25°C and 60 rpm

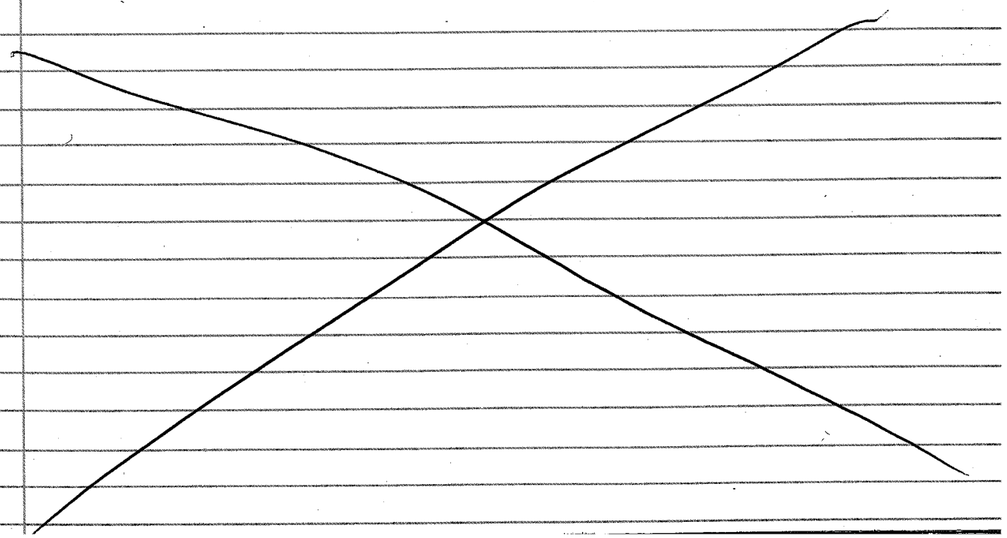


TABLE 1

$NaNO_3$-$CaNO_3$ (0.05 N) Isotherm Experiment

Sample Label $CaNaNO_3$* 0.05*	$E_{i,Ca}$ used	Wt. Zeol. to use (gm)	Vol. Soln. Used (ml)	Wt. Zeol. Used (gm)	Wt. Bottle Empty (gm)	Wt. Bottle + Soln. (gm)	Sample Dilution Factor		Diluted Sample Concentration (ppm)		Reference Dilution Factor		Diluted Reference Concentration (ppm)	
							Na+	Ca++	Na+	Ca++	Na+	Ca++	Na+	Ca++
1	.1	.2448	25	.2449	8.0189	33.1522	1/10	1	103	20.7	10/100	1	98.8	88.2
2	.1	.1493	25	.1494	7.9866	33.0869	1/10	1	115	33.8	-	-		
3	.2	.2880	25	.2883	8.0488	33.3073	1/10	1	110	51.7	1	1	891	169
4	.2	.2033	25	.2032	7.9512	33.0555	1/10	1	108	63.8	-	-		
5	.2	.1557	25	.1557	7.9836	33.1062	1/10	1	104	80.2	-	-		
6	.3	.2057	25	.2056	7.9990	33.1994	1/10	1	3 TD 11/29/01	119	1	1	769	250
7	.4	.2560	25	.2564	8.0542	33.2811	1/10	1	91.6	167	1	1	647	331
8	.4	.1800	25	.1801	7.9972	33.0287	1/10	1	82.5	199	-	-		
9	.5	.2304	25	.2304	8.0228	33.1486	1/10	1	76.8	233	1	1	527	414
10	.6	.2174	25	.2174	7.9524	33.0696	1/10	1	6 TD 11/29/01	301	1	1	421	482
11	.7	.2021	25	.2024	8.0057	33.1157	1/10	1	52.3	376	1	1	316	546
12	.7	.1465	25	TD 11/27/01 1461.1462	8.0292	33.0465	1/10	1	51.7	477	-	-		
13	.8	.1858	25	.1861	7.9672	33.0055	1/10	1	43.7	448	1	1	210	627
14	.9	.2323	25	.2324	7.9213	33.0506	1/10	1	37.8	481	1	1	108	698
15	1.0	.2541	25	.2542	7.9431	33.2198	1/10	1	30.6	524	-	1		763
16	1.0	.2000	25	.2000	7.9348	33.1624	1/10	1	24.7	545	-	-		
17	1.0	.1516	25	.1518	7.9665	33.1505	1/10	1	19.4	590	-	-		
18	1.0	.2134	50	.2136	11.4995	61.6558	1/10	1	14.1	623	-	-		
19	1.0	.1355	50	.1356	11.6759	61.7486	1/10	1	9.54	638	-	-		
20	1.0	.1000	50	.1000	11.4762	61.5023	1/10	1	7.04	653	-	-		
21	1.0	.0640	50	.0640	11.5421	61.5315	1/10	1	4.84	674	-	-		

12/4/01 ROP

Preparation of Zeolite samples to be sent to Texas Tech Univ. for TGA analysis

Procedure: Place ~ 0.5 gm of the zeolite powder in a glass vial. The vials are labeled A to O, and the number of the bottle for the reference TGA. The volume of the vials have VBS stamped on the side. The vials and the corresponding sample identification are given below.

A - CNA*100/200*UC*WA*HL*CAT
B - VBS 7A
C - CNA*100/200*UC*WA*HL*CAT*Naf
D - CNA*100/200*UC*WA*HL*CAT*Caf
E - CNA*100/200*UC*WA*HL*CAT*Kf
F - CNA*100/200*UC*WA*HL*CAT*HF
G - CNA*100/200*UC*WA*HL*CAT*HF-Naf
H - CNA*100/200*UC*WA*HL*CAT*HF-Caf
I - CNA*100/200*UC*WA*HL*CAT*HF-Kf
J - ASH*450/635*UC*WA
K - CNA*100/200*UC*WA*HL*CAT*HF
L - CNA*100/200*UC*WA*HL*CAT*Naf
M - CNA*100/200*UC*WA*HL*CAT*Caf
N - CNA*100/200*UC*WA*HL*CAT*Kf
O - VBS 7A

Samples sent to Bill Shannon of Texas Tech by Federal Express.

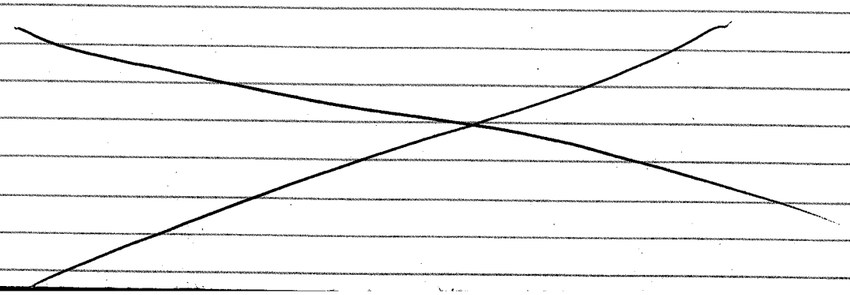
2 Dec 1991 TD

TD 12/5/91

Remeasured $NaCl/CaCl_2$ (.5N) @25°C & solutions

(ORIGINAL MEASUREMENTS IN PAGE 61)

$E_{Ca^{2+}}$	Actual Diluted Na^+ (ppm)
0.02	577
0.04	553
0.06	545
0.08	531
0.1	522
0.12	498
0.15	485
0.2	443
0.25	431
0.3	403
0.4	358
0.5	
0.6	252
0.65	218
0.7	192
0.75	156
0.8	124
0.83	101
0.87	165
0.9	118
0.93	734
0.95	48.1

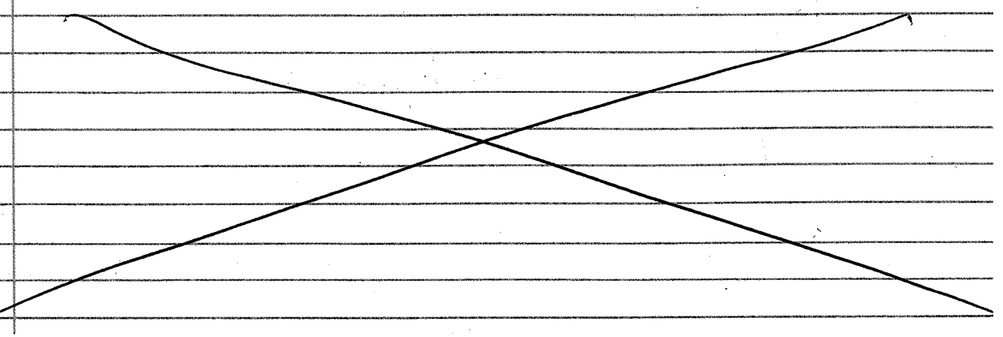


3 Dec 1991 TD

Remeasured $NaCl/CaCl_2$ (0.05N) @25°C solutions

(ORIGINAL MEASUREMENTS ON PAGE 62)

$E_{Ca^{2+}}$	Actual Diluted Na^+ (ppm) ± STD
0.015	113
0.03	114
0.05	106
0.08	95.4
0.1	102
0.15	97.0
0.2	90.5
0.25	84.4
0.3	79.1
0.4	68.6
0.5	57.0
0.55	51.4
0.6	46.9
0.65	42.9
0.7	34.4
0.75	27.5
0.8	21.6
0.85	15.2
0.9	10.6
0.95	5.20



6 DEC 1991 TD 0800

Samples of the $NaNO_3$-KNO_3 (0.05N) isotherm experiment solutions were taken and diluted according to Table 1 on page 151. The appropriate Ionic Strength adjuster was added. The ~~solutions~~ TD 12/6/91 dilutions were put in 15ml pp bottles and labled (eg: $KNaNO_3$ * 0.05 * 1).
Na^+

TD 12/6/91 (EITHER Na^+ or K^+)

6 Dec 1991 TD 1300

Samples of the $NaNO_3$-$Ca(NO_3)_2$ isotherm experiment solution were taken and Diluted according to Table 1 on page 154. The appropriate Ionic Strength adjuster (Either Na^+ or Ca^{2+}) was added. The ~~solutions~~ TD 12/6/91 dilutions were put in 15ml PP bottles and labled (eg: $Ca\,NaNO_3$ * 0.05 * 1
Ca^{2+}).

9 Dec 1991 0930 TD

Na^+ concentration measurements were taken using the Ion Selective electrodes ~~(Meter 920A~~ TD 12/9/91 ORION 920A SERIAL NUMBER 001606) And recorded in Table 1 p151

~~9 Dec 1991 TD 1100~~ TD 12/9/91

The slope for samples 1-16 = 57.2mV

The slope for samples TD 12/9/91 17-21 = 52.4mV

9 DEC 1991 1100 TD

K^+ concentration measurements were taken using the specific Ion selective ~~electrode~~ TD 12/9/91 electrode. (ORION 920A SN 001606) AND recorded in TABLE 1 on p.151

The slope for samples 1-9 = 99.9mV

The slope for samples 10-12 = 44.1mV

The slope for samples 13-15 = 73.3mV

The slope for samples 16-21 = 55.8mV

10 DEC 1991 0930 TD

Na^+ ~~mixture~~ TD Ca^{2+} concentration measurements were taken using the specific Ion Selective Electrode and the ORION 920A (SN 001606) meter and recorded in Table 1 on page 154.

SLOPE for SAMPLES 1-11 = 76.9mV

SLOPE for SAMPLES 12-21 = 52.2mV

10 DEC 1991 1130 TD

Ca^{2+} concentration measurements were taken using the specific Ion Selective Electrode and the ORION 920A (SN001606) Meter and recorded in Table 1 on page 154

SLOPE FOR SAMPLES 1-6 = 30.5 mV

SLOPE FOR SAMPLES 7-11 = 29.5mV

SLOPE FOR SAMPLES 12-21 = 29.2mV

12/18/91 1400 hrs JP

Remeasured NaCl/KCl 0.5N solutions for Na^+ (Original measurements recorded on pages p.52 and p.57)

Reference Solutions 0.5N

E_{i,K^+}	Na^+ (ppm)
.1	525
.2	458
.3	806
.4	689
.5	580
.6	464
.7	346
.9	120

Samples 0.5N

E_{K^+}	Na^+ (ppm)
0.01	573
0.02	559
0.03	555
0.05	543
0.075	525
0.09	516
0.1	514
0.13	494
0.15	481
0.18	477
0.2	453

E_{K^+}	Na^+ (ppm)
0.24	433
0.3	801
0.35	739
0.4	675
0.5	553
0.6	450
0.7	323
0.75	274
0.8	220
0.9	109

12/18/91 0830 hrs JP

Remeasured NaCl/KCl 0.05N solutions for Na^+ (original measurements recorded on pages 53 and 58

0.5N Reference Solutions

E_{i,K^+}	Na^+ (ppm)
.1	101
.2	911
.3	791
.4	689
.7	343

0.5N Samples

E_{K^+}	Na^+ (ppm)
0.01	114
0.02	112
0.03	115
0.05	109
0.075	105
0.09	103
0.1	102
0.13	98.4
0.15	95.4
0.18	93.5
0.2	90.6

E_{K^+}	Na^+ (ppm)
0.24	85.1
0.3	78.9
0.35	71.8
0.4	66.6
0.5	56.8
0.6	44.5
0.7	33.3
0.75	28.3
0.8	22.7
0.9	11.3

1030hrs JP

Remeasured NaCl/KCl 0.005N solutions for Na^+ (original measurements recorded on pages 53 and 59).

0.005N Reference Solutions

E_{i,K^+}	Na^+ (ppm)
.2	91.7
.3	85.5
.4	68.9
.5	57.9
.6	47.1
.8	24.4
.9	12.5

0.005N Samples

E_{K^+}	Na^+ (ppm)	E_{K^+}	Na^+ (ppm)
0.01	113	0.24	83.8
0.02	112	0.3	76.9
0.03	111	0.35	71.1
0.05	109	0.4	66.2
0.075	107	0.5	57.7
0.09	104	0.6	44.5
0.1	102	0.7	39.9
0.13	96.5	0.75	28.3
0.15	94.3	0.8	22.5
0.18	92.5	0.9	11.4
0.2	88.9		

12/20/91 0900hrs JP

Reference solutions for the $NaNO_3$-$Ca(NO_3)_2$ isotherm experiment were taken and diluted according to Table 1 on p 154. The solutions were placed in 60 ml PP or 125ml PP bottles and labeled (eg $CaNaNO_3$ * 0.05N * .1 X_{Ca}, Ca^{++}). .2 ml of ionic strength adjustor for Ca^{++} analysis was added to each bottle.

Reference solutions for the $NaNO_3$ - KNO_3 (0.05N) isotherm experiments were taken and diluted according to Table 1 on p 151. Solutions were placed in either 60 ml or 125 ml PP bottles and labeled (eg. $KNaNO_3$ * 0.05N * .1 X_{K^+}, K^+).

1300hrs JP

Ca^{++} concentration measurements were taken for the $CaNaNO_3$ * 0.05N reference solutions. Results are recorded in Table 1 on p154.

Slope = 28.7 mV

12/23/91 0900hrs JP

Appropriate ISA was placed in all 60ml bottles containing reference solutions (1.0 ml for Na^+ analysis of $KNaNO_3$ and $CaNaNO_3$ references at .05N and 0.2ml of ISA for K^+ and Ca^{++} analysis of $KNaNO_3$ and $CaNaNO_3$ references at 0.5N). Reference solutions in 125 ml bottles do not contain ISA. For analysis of these solutions take 10 ml and add ISA before measurement.

12/23/91 0945 TD

TD 12/23/91

Remeasurement of ~~K^+~~

Remeasured NaCl/KCl 0.5N solutions for K^+ (original measurements on page 52 & 57)

TD 12/23/91

E_{K^+}
0.01
0.02
0.03
0.05
0.075
0.09
0.1
0.13
0.15
0.18
0.2
0.24
0.3
0.35
0.4
0.5
0.6
0.7
0.75
0.8
0.9

12/23/91 0945 TD

TD 12/23/91

Remeasured NaCl/$CaCl_2$ 0.5N solutions for Ca^{++} (original measurements recorded on pages 54 and 61)

$E_{Ca^{2+}}$	Ca^{2+} (ppm)
0.02	25.9
0.04	50.6
0.06	67.2
0.08	95.2
0.1	124
0.12	141
0.15	164
0.2	207
0.25	256
0.3	319
0.4	435
0.5	551
0.6	652
0.65	708
0.7	769
0.75	822
0.8	908
0.83	946
0.87	494
0.9	516
0.93	529
0.95	542

SLOPES

.02 → .3 28.8mV

.4 → .95 28.7mV

REFERENCE SOLUTIONS

$E_{Ca^{2+}}$	Ca^{2+} (ppm)
0.1	100
0.2	208
0.3	313
0.4	422
0.5	528
0.6	626
0.7	737
0.8	829
0.9	969
1.0	1060

SLOPE: 28.9 mV

12/27/91 0930 TD

Remeasured NaCl/$CaCl_2$ 0.05N solutions for Ca^{2+} (original values ~~are~~ TD 12/27/91 recorded on pages 55 & 62)

$E_{Ca^{2+}}$	Ca^{2+} (ppm)
0.015	21.8
0.03	41.0
0.05	68.1
0.08	90.9
0.1	113
0.15	172
0.2	224
0.25	275
0.3	333
0.4	438
0.5	546
0.55	612
0.6	675
0.65	733
0.7	808
0.75	868
0.8	932
0.85	985
0.9	1040
0.95	1110

SLOPE = 28.9 mV

Reference Solutions

$E_{Ca^{2+}}$	Ca^{2+} (ppm)
0.1	120
0.2	227
0.3	349
0.4	467
0.5	583
0.6	715
0.7	834
0.8	977
0.9	1070
1.0	1350

SLOPE = 28.9 mV

12/27/91 1110 TD

Remeasured NaCl/$CaCl_2$ .005 N solutions for Ca^{2+} (original values ~~are~~ were TD 12/27/91 recorded on pages 55 & 63)

$E_{Ca^{2+}}$	Ca^{2+} (ppm)
0.01	2.46
0.03	3.63
0.05	6.96
0.08	8.92
0.1	11.2
0.13	14.9
0.17	20.5
0.2	24.9
0.23	25.3
0.27	29.8
0.3	33.3
0.35	36.8
0.4	39.8
0.45	57.0
0.5	61.2
0.6	69.9
0.65	73.3
0.7	83.0
0.75	86.4
0.8	92.7
0.85	98.6
0.9	103
0.95	109

SLOPE = 29.5 mV

Reference Solutions

$E_{Ca^{2+}}$	Ca^{2+} (ppm)
.3	39.5
.4	51.9
.5	64.2
.6	76.2
.7	86.4
.8	98.6
.9	22
1.0	126

The 0.1 & 0.2 solutions could not be located.

SLOPE = 29.5 mV

12/23/91 1330 T.6.

Remeasured all NaCl/KCl solutions from the 25°C isotherm experiment except those w/ K^+ stds.

Purpose: To check the reproducibility of earlier Na^+ measurements.

* This data corresponds with pg 98 and was actually measured on 8/5/91

NaCl/KCl (@ 25°C)

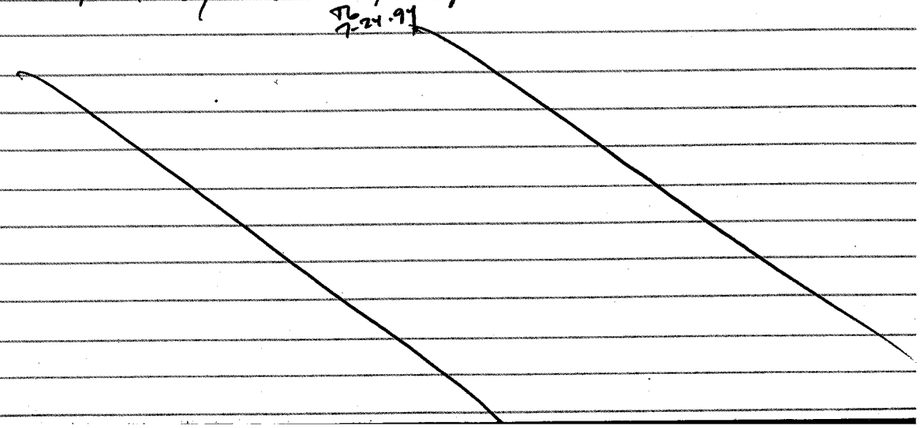
0.5 N		0.05 N		0.005 N	
E_{K^+}	Na^+ ions (ppm)	E_{K^+}	Na^+ ions (ppm)	E_{K^+}	Na^+ ions (ppm)
.01	575	.01	114	.01	114
.02	563	.02	114	.02	114
.03	559	.03	114	.03	114
.05	542	.05	109	.05	110
.075	528	.075	106	.075	106
.09	518	.09	106	.09	104
.1	509	.1	103	.1	105
.13	491	.13	99.6	.13	99.2
.15	478	.15	97.3	.15	97.2
.18	452	.18	93.9	.18	93.9
.2	450	.2	92.0	.2	914
.24	424	.24	86.7	.24	84.7
.3	801	.3	81.3	.3	79.2
.35	731	.35	73.3	.35	72.5
.4	675	.4	66.7	.4	66.8
.5	555	.5	56.6	.5	56.5
.6	445	.6	45.0	.6	44.1
.7	321	.7	33.4	.7	34.0
.75	267	.75	27.8	.75	28.0
.8	213	.8	22.1	.8	22.6
.9	103	.9	11.2	.9	11.0

Cont. Average slope of 0.5N Na^+ electrode: 57.9 mV
Average slope of 0.05N Na^+ electrode: 58.4 mV
Average slope of 0.005N Na^+ electrode: 58.9 mV

NaCl/KCl References (@ 25°C)

0.5 N		0.05 N		0.005 N	
E_{i,K^+}	Na^+ ions (ppm)	E_{i,K^+}	Na^+ ions (ppm)	E_{i,K^+}	Na^+ ions (ppm)
0.1	517	0.1	105	0.1	—
0.2	465	0.2	923	0.2	93.9
0.3	811	0.3	804	0.3	82.1
0.4	693	0.4	695	0.4	71.2
0.5	587	0.5		0.5	58.5
0.6	471	0.6		0.6	47.1
0.7	354	0.7	342	0.7	35.4
0.8	—	0.8		0.8	23.6
0.9	122	0.9	110	0.9	12.1
1.0	—	1.0	—	1.0	—

Note: None of these results have been multiplied by their ~~cap~~ respective dilution factor.



12/23/91 T.G 1400

Preparation of $NaHCO_3/KHCO_3$ (0.5N) Solutions

Objective - To prepare various $NaHCO_3/KHCO_3$ solutions, all of which have a total normality of 0.5.

Method - Weigh the compounds using the previously calculated weights which were predicted before the experiment

Equipment - Mettler AE240, balance accurate to .001g (serial # 101237)
weighing bottle(s)
metal spatula(s)
100 ml beaker(s)
250 ml volumetric flask(s)
eye dropper
corning stirrer & magnets & magnetic rod
17 - 250 ml Nalgene polypropylene bottles

Reagents - Sodium Bicarbonate Lot# 897186 A
Potassium Bicarbonate Lot# 912932
Ultrapure Water (>17 MΩ resistivity)

Procedure -

Preparation of $NaHCO_3$-$KHCO_3$ Solutions

Written by: R. T. Pabalan **Date:** November 19, 1991
Revision No.: 0 **Date Revised:**

1. Prepare 250 ml of $NaHCO_3$-$KHCO_3$ aqueous mixture with a total normality of 0.5 N and a fixed Na/K ratio by taring reagent grade $NaHCO_3$ and $KHCO_3$ in the amounts given in Table 1 onto a clean (acid-washed) 250 ml beaker. Keep the beaker covered with parafilm between additions of reagents to minimize adsorption of water from the atmosphere. Add about 100 ml ultrapure water (>17 megaohm resistivity) to the beaker, add a stir bar and dissolve the salts completely on a stir plate (add more water if necessary). Retrieve the stir bar with a magnetic rod, then carefully rinse the rod and stir bar with ultrapure water, making sure to catch all the washings in the beaker.

Decant the solution into a clean 250 ml volumetric flask (with the help of a glass rod); rinse the beaker (and glass rod) with ultrapure water several times, carefully transferring all washings into the volumetric flask. Then fill the flask to about 2-3 inches below the mark with ultrapure water; swirl or shake the flask. Let stand for a few seconds, then add water dropwise up to the mark; remix.

Transfer the solution into a clean 250 ml polypropylene bottle. Label the bottle (e.g., $NaHCO_3/KHCO_3$*0.5N*0.1X_K, plus Date and Initial).

TABLE I.

X_K (0.5 N) ($NaHCO_3$-$KHCO_3$ soln.)	Wt. $NaHCO_3$ needed for 250 ml	Wt. $KHCO_3$ needed for 250 ml	Wt. $NaHCO_3$ used	Wt. $KHCO_3$ used
0.01	10.396	0.12514	10.39613	.12519
0.025	10.238	0.31286	10.23821	.31281
0.05	9.9757	0.62571	9.97586	.62570
0.075	9.7132	0.93857	9.71318	.93851
0.1	9.4507	1.2514	9.45066	1.25165
0.2	8.4006	2.5029	8.40069	2.50286
0.3	7.3505	3.7543	7.35069	3.75473
0.4	6.3005	5.0057	6.30078	5.00560
0.5	5.2504	6.2571	5.25055	6.25738
0.6	4.2003	7.5086	4.20047	7.50849
0.7	3.1502	8.76	3.15029	8.76007
0.8	2.1002	10.011	2.10022	10.01097
0.9	1.0501	11.263	1.05032	11.26308
0.925	0.78756	11.576	.78763	11.57643
0.95	0.52504	11.889	.52515	11.88925
0.975	0.26252	12.201	.26265	12.20128
1	0	12.514	—	12.51423

12/23/91 dwP
Received by first class mail
results of chemical analyses of
samples sent to Texas Tech Univ
on 12/4/91 (see page GE-04-135),
a copy of the results are shown below

ANALYTICAL SERVICES LABORATORY
Department of Geosciences
Texas Tech University

INVOICE

Date 18-Dec-91

Number 91107

PO # 14578

To:
Southwest Research Institute
P.O. Box 28510
6220 Culebra Road
San Antonio, TX 78228

Attention: Bobby Pabalan

SERVICES RENDERED	QUANTITY	UNIT COST	EXTENDED COST
Crushing (<5 grams) with agate mortar and pestle	15	$5.00	$75.00
Fusion with LiBO2; dissolution in HCl	15	$5.00	$75.00
ICP analysis of major elements (Oxides of Si, Ti, Al, Fe, Mn, Mg, Ca, Na, K, P)	15	$10.00	$150.00
ICP analyis of trace elements (Ba, Sr, Sc, Y, Zr, Nb, V, Cr, Ni, Cu, Zn; Rb by AA)	15	$10.00	$150.00

Subtotal of Services $450.00
Rush Service Charge $450.00

TOTAL $900.00

Samples Recieved 12/09/91

Remit Payment to:

TEXAS TECH UNIVERSITY
DEPARTMENT OF GEOSCIENCES
PO Box 41053
LUBBOCK, TX 79409-1053

Attention: Barbara Graham

THANKS FOR YOUR BUSINESS!

William M. Shannon
12/19/91

Contract work for Bobby Pabalan, SRI
Sample fusions by John P. Schnable 12/15/91 - 12/17/91
Majors analyzed (ICP) by William M Shannon 12/17/91
Traces analyzed (ICP) by William M Shannon 12/18/91
Rb analyzed (AA) by William M. Shannon 12/18/91
Reported out by William M. Shannon 12/18/91

Standard values used For TTU ICP calibrations, (*)=Majors, (**)=Trace

Standard=>	.5MHA	.5ABA	.5TMS	ABA	BCM	TMS	MHA	GSP-1	RGMT	1.5ABA	1.5BCM	1.5TMS	2GSP-1
Source==>>	TTU	TTU	TTU	TTU	TTU	TTU	TTU	USGS	TTU	TTU	TTU	TTU	USGS
.(Wt%)	*/**		**	**	**	*	*		*/**	**		*	
SiO2	38.42	22.98	29.84	45.8	55.16	59.68	60.84	67.32	72.87	68.7	82.74	89.52	134.64
TiO2	0.43	1.85	0.86	2.89	1.11	0.12	0.85	0.66	0.27	3.135	1.67	0.18	1.32
Al2O3	8.76	7.18	9.02	14.36	15.85	18.04	17.51	15.28	13.61	21.54	23.78	27.06	38.56
Fe2O3Tot^	3.18	6.14	2.78	12.27	8.46	5.4	6.2	4.3	1.85	18.485	12.69	8.18	8.68
MnO	0.05	0.18	0.12	0.19	0.14	0.24	0.1	0.04	0.04	0.285	0.21	0.36	0.88
MgO	1.57	5.67	0.06	11.34	5.48	0.12	3.13	0.97	0.3	17.01	0.22	0.18	1.94
CaO	3.06	5.31	0.62	10.61	7.07	1.24	6.11	2.03	1.25	15.915	10.61	1.06	4.06
Na2O	2.14	1.53	4.56	3.05	3.06	9.12	4.28	2.81	4.1	4.575	5.79	13.68	5.62
K2O	0.68	0.71	2.09	1.41	2.09	4.18	1.19	5.51	4.24	2.115	3.14	6.27	11.02
P2O5	0.09	0.23	0.08	0.46	0.45	0.15	0.18	0.28	0.07	0.69	0.68	0.23	0.56
Total	58.28	58.79	49.15	101.58	99.67	98.29	100.39	99.2	98.6	152.37	149.51	147.44	198.48
(ppm)													
Rb	9.5	13.5	60	27	34	120	19	254	150	40.5	51	180	500
Sr	274	269	372	537	719	744	548	233	110	806	1079	1116	466
Ba	153	202	310	484	909	620	306	1300	893	606	1364	930	2600
Sc	6.5	14.5	0.3	29.1	18.7	0.5	12.9	6.6	4.6	43.6	28.1	0.8	13.2
Ti	2548	6265	360	12530	6655	719	5096	3990	1619	18795	9982	1079	7980
V	51	110	0	220	137	0	102	54	12	330	206	0	100
Cr	24	202	2	404	157	5	49	12	2	606	236	7	24
Mn	387	736	929	1471	1084	1859	774	331	310	2207	1626	2788	662
Ni	17	125	2	250	94	5	35	12	1	375	141	7	24
Cu	9	25	0	50	46		18	33		75	70		66
Zn	33	44	112	88	72	224	65	98	32	132	108	336	196
Nb	6	24	166	48	34	332	11	29	11	72	51	498	50
Y	8.0	14.9	24.7	29.7	22.2	49.4	16.0	30.4	26.1	44.6	33.3	74.1	60.8
Zr	72	77	602	153	147	1204	145	500	223	230	220	1806	1000

TTU standards analyzed during majors run. (GSPT is resampled GSP-1)

Sample==>>	2.0GSPT	GSPT	RGMT	1.5TMS	MHA	BLANK	TMS	0.5MHA
Repeats==>	(3)	(5)	(2)	(5)	(4)	(5)	(4)	(1)
(Wt%)								
SiO2		69.00	75.03	89.00	61.40	-0.05	60.21	31.07
TiO2	0.13	0.07	0.03	0.02	0.00	-0.00	0.01	0.04
Al2O3	30.09	15.23	13.90	27.10	17.50	-0.02	10.22	0.09
Fe2O3Tot^	0.92	4.67	1.99	0.09	6.34	-0.03	5.54	3.15
MnO	0.09	0.05	0.04	0.36	0.11	-0.00	0.25	0.05
MgO	1.94	0.99	0.30	0.15	3.14	0.03	0.11	1.57
CaO	4.30	2.15	1.25	1.06	6.16	0.02	1.23	3.07
Na2O	5.67	2.00	4.22	13.05	4.31	-0.02	9.09	2.13
K2O	.10.44	5.40	4.32	6.20	1.24	-0.01	4.17	0.62
P2O5	0.56	0.31	0.05	0.25	1.11	0.01	0.16	0.00
Total%	62.13	100.91	101.12	147.06	101.40	-0.07	90.90	50.60
(ppm)								
Sr	489	244	109	1121	559	-1.30	736	270
Ba		1404	099	934	317	-2.09	609	155
Y	60.4	30.2	25.7	74.5	17.1	-0.14	50.0	0.1
Zr	1159	624	223	1004	141	0.12	1215	74

TTU standards analyzed during trace run.

Sample==>>	2.8GSPT	GSPT	RGMT	1.5TMS	1.5BCM	BLANK	BCM	0.5MHA
Repeats==>	(1)	(2)	(2)	(1)	(1)	(2)	(2)	(2)
(ppm)								
Rb	498	241	150	181	51.9		32.6	9.8
Sr	467	240	188	1046	1058	-1.49	695	278
Ba	2863	1428	876	899	1373	-0.98	900	156
Sc	11.8	6.2	4.5	0.6	27.5	0.01	18.7	6.5
V	90	53	13	<2	184	-1.01	134	51
Cr	21	12	3	2	232	1.09	157	23
Ni	15	8	2	4	131	-0.92	97	17
Cu	58	27	8	8	89	0.11	47	11
Zn	160	100	34		102	-0.54	75	36
Nb	58	33	11		45	0.40	33	4
Y	66.8	30.5	26.0		31.8	0.07	21.3	7.9
Zr	1002	567	219		73	-0.49	148	73

Sample==>>	SRI-A	SRI-B	SRI-C	SRI-D	SRI-E	SRI-F	SRI-G	SRI-H	SRI-I	SRI-J	SRI-K	SRI-L
ICP#	1175	1176+1225	1182	1183	1185+1223	1189	1191	1193	1195+1203	1207	1209	1212
(Wt%)												
SiO2	69.01	68.91	68.44	67.58	67.30	74.32	72.72	72.54	68.88	53.77	70.67	66.23
TiO2	0.01	0.00	0.01	0.01	0.01	0.01	0.01	0.01	0.01	0.00	0.01	0.01
Al2O3	11.39	17.86	11.24	11.14	11.10	10.22	10.76	10.33	11.20	23.29	9.81	10.94
Fe2O3Tot^	0.46	0.07	0.37	0.40	0.41	0.36	0.44	0.41	0.39	0.11	0.39	0.42
MnO	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00
MgO	0.29	0.07	0.18	0.24	0.16	0.22	0.26	0.24	0.09	0.03	0.28	0.24
CaO	0.12	0.13	0.04	5.85	0.04	0.13	0.13	0.12	0.03	0.01	0.09	5.88
Na2O	4.00	2.54	6.38	0.01	0.01	0.17	1.57	0.64	6.32	14.47	0.24	0.03
K2O	2.70	11.16	0.67	0.43	9.70	2.19	2.56	2.45	0.67	0.01	2.17	0.49
P2O5	0.02	0.01	0.02	0.01	0.01	0.00	0.01	0.01	0.01	0.01	0.01	0.01
Total%	88.79	100.74	87.34	85.66	88.74	87.61	88.45	86.75	87.68	91.69	83.59	84.25
(ppm)												
Sr	55	65	6	95	3	49	55	52	7	7	50	95
Ba	238	124	132	165	5	221	241	241	133	3	226	167
Y	8.8	2.1	8.7	8.4	8.7	4.4	6.3	4.3	8.8	0.5	3.2	8.3
Zr	89	1	86	87	84	92	91	91	86	1	88	82

Sample==>>	SRI-A	SRI-B	SRI-C	SRI-D	SRI-E	SRI-F	SRI-G	SRI-H	SRI-I	SRI-J	SRI-K	SRI-L
ICP#	1256	1257	1268	1262	1264	1267	1268	1270	1271	1273	1274	1276
(ppm)												
Rb	97	527	41	29	25	91	99	98	41	27	94	29
Sr	56	65	7	95	3	49	56	53	7	5	49	96
Ba	239	121	133	160	6	207	243	239	133	2	221	164
Sc	1.3	0.1	1.2	1.1	1.2	1.2	1.1	1.1	1.1	0.0	1.0	1.2
V	<1	<1	2	2	2	2	<1	<1	<1	2	<1	<1
Cr	2	2	2	4	4	4	3	2	2	159	14	2
Ni	3	38	4	3	3	1	3	1	4	77	9	4
Cu	5	13	4	2	2	1	3	1	3	5	2	1
Zn	12	1	12	8	8	8	12	9	8	<1	7	7
Nb	15	1	15	16	16	18	14	16	15	1	15	15
Y	8.1	1.3	7.5	7.6	7.8	3.8	5.8	4.8	7.7	0.2	2.9	7.7
Zr	87	2	84	81	85	90	88	86	84	2	88	84

Sample==>>	SRI-M	SRI-N	SRI-O	SRI-B	SRI-B	SRI-E	SRI-E	SRI-I	SRI-I	SRI-M	SRI-M
ICP#	1214+1222	1216	1218	1176	1225	1185	1223	1195	1203	1214	1222
(Wt%)											
SiO2	67.29	68.72	64.96	69.33	68.50	68.14	66.47	70.18	67.58	67.37	67.21
TiO2	0.01	0.01	0.00	<0.01	0.00	0.01	0.01	0.01	0.01	0.01	0.01
Al2O3	11.12	11.42	20.15	17.91	17.80	11.23	10.97	11.48	10.99	11.14	11.10
Fe2O3Tot^	0.42	0.41	0.07	0.05	0.09	0.39	0.42	0.38	0.40	0.42	0.43
MnO	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00
MgO	0.17	0.17	0.05	0.07	0.07	0.16	0.16	0.18	0.00	0.17	0.18
CaO	0.04	0.02	2.11	0.14	0.12	0.06	0.03	0.04	0.01	0.04	0.04
Na2O	0.02	6.43	6.29	2.50	2.58	<0.01	0.03	6.29	6.34	0.03	0.02
K2O	9.70	0.74	5.20	11.01	11.30	9.68	9.71	0.66	0.68	9.67	9.72
P2O5	0.01	0.01	0.02	0.01	0.01	0.01	0.01	0.01	0.01	0.01	0.01
Total%	88.78	87.92	98.85	101.02	100.47	89.66	87.81	89.16	86.03	88.84	88.71
(ppm)											
Sr	4	8	447	64	66	2	4	6	8	4	4
Ba	7	135	2861	121	127	2	7	131	134	7	7
Y	9.2	9.3	1.9	1.7	2.6	8.4	9.8	8.7	8.9	8.8	9.7
Zr	84	86	7	2	<1	84	85	88	83	85	84

Sample==>>	SRI-M	SRI-N	SRI-O
ICP#	1277	1281	1282
(ppm)			
Rb	27	41	111
Sr	2	7	437
Ba	7	131	2755
Sc	1.2	1.1	0.2
V	2	<1	1
Cr	1	2	2
Ni	0	1	18
Cu	1	2	6
Zn	11	7	4
Nb	15	15	3
Y	7.6	7.4	1.6
Zr	83	86	8

Bobby, We got your samples 12/5/91 and have sent you the results by mail and Fax 12/19/91. Rb was done by AA. The majors run was optimized for maximum range in alkali content. The trace run was optimized for linearity at the low end. Call me if you have questions WM Shannon

12/27/91 T.G. 0900 cont. to pg 182

NaCl/KCl Reverse Isotherm Experiment

NaCl/$CaCl_2$ " " "

Objective - to observe an equilibrium shift of the isotherm in the desired direction; to prove reversibility of cation exchange.

Method - by increasing the cation concentration for one of the two elements in the experiment, since we know the original volume and concentrations, we can then add another known volume and concentration and observe where equilibrium occurs for the cation exchange in the CDV.

Reagents - The reagents were the $0.1X_K$ & $1.0X_K$ and $0.1X_{CA}$ & $1.0X_{CA}$ for each of the different normality solutions.
∴ a total of 11 solutions were used, since $.1X_{CA}$ (.005 N) wasn't used.

See pg 27-32 & 44-45 for the following soln preparations

0.5N: $0.1X_K$, $1.0X_K$, $0.1X_{CA}$, & $1.0X_{CA}$

0.05N: $0.1X_K$, $1.0X_K$, $0.1X_{CA}$, & $1.0X_{CA}$

0.005N: $0.1X_K$, $1.0X_K$, & $1.0X_{CA}$

Procedure - 1.) Obtain the original samples from pg 36-49
2.) Using every other CDV -- NaCl/KCl*$CaCl_2$ soln, add a predetermined volume of one of the above reagents to its respective CDV mixture

using the following charts.
3.) Allow mixtures to come to equilibrium and measure the concentrations.

NaCl/KCl (0.5N) Reverse Exp't.

Soln #	E_{K^+}	$K_{Na,K}$	Equil. conc. (ppm) Na^+	Equil. conc. (ppm) K^+	Equil. soln vol. (ml)	Vol. - new Na/K added (ml)	Measured ppm of Na/K added Na^+	Measured ppm of Na/K added K^+	Calcd standard conc. (ppm) Na^+	Calcd standard conc. (ppm) K^+	Sample Dilution factor Nat	Sample Dilution factor K^+
①	0.01	1.0	11480	1165	8.5	3	0	19310	8485	5124	0.5/10	1/10
②	0.03	1.0	11260	5045	8.5	3	0	19310	8323	5410	0.5/10	1/10
③	0.075	1.0	10640	14685	8.5	3	0	19310	7864	6135	0.5/10	1/10
④	0.10	1.0	10440	19155	8.5	3	0	19310	7717	6453	0.5/10	1/10
⑤	0.15	1.0	9760	30105	8.5	5	0	19310	6145	9047	0.5/10	1/10
⑥	0.20	1.0	9160	38855	8.5	5	0	19310	5767	9598	1/10	1/10
⑦	0.30	1.0	8040	52855	8.0	5	0	19310	4948	11612	1/10	1/10
⑧	0.40	1.0	6880	72650	8.0	5	10360	19000	4218	15438	0.5/10	0.5/10
⑨	0.60	1.0	4610	117400	8.5	5	10360	19000	6740	8096	0.5/10	0.5/10
⑩	0.75	1.0	2760	144100	8.5	5	10360	19000	5575	9814	1/10	1/10

NaCl–KCl (0.05 N) Reverse Expt.

Soln # / E_{K^+}	Equilib. ppm Na+	Equilib. ppm K+	Equilib. Soln Vol. (ml)	Vol. new Na/K added (ml)	Measured ppm of Na/K added: Na+	Measured ppm of Na/K added: K+	Calcd Starting ppm of: Na+	Calcd Starting ppm of: K+	Sample Dilution Factor: Na+	Sample Dilution Factor: K+	E_{i,K^+}
(1) 0.01	1110	9.69	14	5	0 ($E_{K,i}$ = 1.0)	1945	819	519	1/10	1	1.0
(3) 0.03	1120	45.0	14	5	0	1945	825	545	1/10	1	1.0
(5) 0.075	1060	143	14	10	0	1945	618	894	1/10	1	1.0
(7) 0.10	1040	196	14	10	0	1945	607	925	1/10	1	1.0
(9) 0.15	980	303	14	10	0	1945	572	987	1/10	1	1.0
(11) 0.20	920	389	14	10	0	1945	537	1037	1/10	1	1.0
(13) 0.30	788	589	14	10	0	1945	460	1154	1/10	1	1.0
(15) 0.40	672	816	14	10	1060 ($E_{K,i}$ = 0.1)	199	834	559	1/10	1	0.1
(17) 0.60	447	1135	23	5	1060	199	556	968	1/10	1	0.1
(19) 0.75	271	1460	23	5	1060	199	412	1235	1/10	1	0.1

NaCl–KCl (0.005 N) Reverse

Soln. # / E_{K^+}	Equilib. ppm Na+	Equilib. ppm K+	Equil. Soln. Vol. (ml)	Vol. New Na/K added (ml)	Measured ppm of Na/K added: Na+	Measured ppm of Na/K added: K+	Calcd Starting ppm of: Na+	Calcd Starting ppm of: K+	Sample Dilution Factor: Na+	Sample Dilution Factor: K+	E_{i,K^+}
(1) 0.01	11.4	1.03	30	20	0 ($E_{K,i}$ = 1.0)	188	6.84	75.8	1	1	1.0
(3) 0.03	11.3	4.46	30	20	0	188	6.79	77.9	1	1	1.0
(5) 0.075	10.7	14.6	30	10	0	188	80.3	58.0	1	1	1.0
(7) 0.10	10.3	20.8	30	10	0	188	77.3	62.6	1	1	1.0
(9) 0.15	9.57	32.3	30	10	0	188	71.8	71.2	1	1	1.0
(11) 0.20	9.05 low ~10 ml	41.4	30	10	0	188	67.9	78.1	1	1	1.0
(13) 0.30	78.1	61.6	30	10	0	188	59.6	93.2	1	1	1.0
(15) 0.40	65.3	78.4	30	10	103 ($E_{K,i}$ = 1.0)	19.5	74.7	63.7	1	1	1.0
(17) 0.60	44.2	116	80	20	103 ($E_{K,i}$ = 0.1)	19.5	56.0	96.7	1	1	0.1
(19) 0.80	32.0	156	230	20	103	19.5	28.5	145	1	1	0.1

NaCl – $CaCl_2$ (0.5 N) Reverse Exptl.

Soln # / E_{Ca}^{2+}	Equil. ppm Na+	Equil. ppm Ca++	Equil. Soln Vol. (ml)	Vol. new Na/Ca added (ml)	Measured ppm of Na/Ca added Na+	Measured ppm of Na/Ca added Ca++	Calcd Starting ppm of Na+	Calcd Starting ppm of Ca++	Sample Dilution Factor Na+	Sample Dilution Factor Ca++	$E_{i,Ca}$
① 0.02	11400	237	3.5	2	0 ($E_{Ca,i}$ = 1.0)	10100	7255	3824	0.5/10	0.5/10	1.0
③ 0.06	10700	667	3.5	2	0	10100	6809	4097	0.5/10	0.5/10	1.0
⑤ 0.10	10280	1240	3.5	2	0	10100	6542	4443	0.5/10	0.5/10	1.0
⑦ 0.15	9700	1610	3.5	3	0	10100	6223	5528	0.5/10	0.5/10	1.0
⑨ 0.25	8560	2430	3.5	3	0	10100	4609	5880	0.5/10	0.5/10	1.0
⑪ 0.40	6820	3880	3.5	5	0	10100	2908	7543	1/10	1/10	1.0
⑬ 0.60	4520	5900	3.5	5	0	10100	1861	9371	1/10	1/10	1.0
⑮ 0.70	3480	6980	3.5	5	10400 ($E_{Ca,i}$ = 0.1)	953	7551	3435	1/10	1/10	0.1
⑰ 0.80	2320	8140	3.5	5	10400	953	7073	3912	1/10	1/10	0.1
⑲ 0.87	1500	8460	8.5	5	10400	953	4796	5680	1/10	1/10	0.1

NaCl – $CaCl_2$ (0.05 N) Reverse Exptl.

Soln # / E_{Ca}^{2+}	Equil. ppm Na+	Equil. ppm Ca++	Equil. Soln Vol. (ml)	Vol. new Na/Ca added (ml)	Measured ppm of Na/Ca added Na+	Measured ppm of Na/Ca added Ca++	Calcd Starting ppm of Na+	Calcd Starting ppm of Ca++	Sample Dilution Factor Na+	Sample Dilution Factor Ca++	$E_{i,Ca}$
① 0.015	2130	29.4	14	5	0 ($E_{Ca,i}$ = 1.0)	1020	833	293	1/10	1	1.0
③ 0.05	1090	56.1	14	5	0	1020	803	310	1/10	1	1.0
⑤ 0.10	1040	102	14	10	0	1020	607	485	1/10	1	1.0
⑦ 0.20	920	203	14	10	0	1020	537	543	1/10	1	1.0
⑨ 0.30	810	295	14	10	0	1020	473	597	1/10	1	1.0
⑪ 0.50	580	488	14	10	0	1020	338	710	1/10	1	1.0
⑬ 0.60	460	587	14	10	1030 ($E_{Ca,i}$ = .1)	94.4	698	382	1/10	1	0.1
⑮ 0.70	350	694	14	10	1030	94.4	633	444	1/10	1	0.1
⑰ 0.80	230	804	14	10	1030	94.4	563	508	1/10	1	0.1
⑲ 0.90	120	917	39	10	1030	94.4	306	749	1/10	1	0.1

Reverse Ex pot (+.005V)

Na Cl — Ca Cl2 @ 10 sec

Sample #	Buried 1 mm Na/Ca/Cl cathode			Buried 1 mm Na/Ca/Cl cathode			Sample Dilution Factor			$\zeta_{i,Ca^{2+}}$
	K_{ex}	Ca^{++}	Na^+	K_{ex}	Ca^{++}	Na^+	Ca^{++}	Na^+		
	(mV)	(ppm)	(ppm)	(mV)	(ppm)	(ppm)				
0.0	25	1.94	114	0	99.2	58.4	1	1		1.0
0.1	25	2.82	107	0	99.2	58.4	1	1		1.0
0.2	25	3.71	95.6	0	99.2	58.4	1	1		1.0
0.3	20	4.60	90.3	0	99.2	58.4	1	1		1.0
0.4	20	5.49	82.8	0	99.2	58.4	1	1		1.0
0.5	20	6.38	74.4	0	99.2	58.4	1	1		1.0
0.6	20	7.27	69.2	0	99.2	58.4	1	1		1.0
0.7	20	8.16	62.9	0	99.2	58.4	1	1		1.0
0.8	20	9.05	57.8	0	99.2	58.4	1	1		1.0
0.9	20	9.94	53.5	0	99.2	58.4	1	1		1.0

12/30/91 1030 TD

Remeasured NaCl/KCl 0.5N solutions for K^+
(original values ~~are~~ were recorded on pages 52 and 57)
TD 12/30/91

E_{K^+}	K^+ (ppm)
0.01	10.7
0.02	25.3
0.03	49.6
0.05	92.3
0.075	150
0.09	178
0.10	198
0.13	252
0.15	298
0.18	345
0.2	383
0.24	450
0.3	564
0.35	647
0.4	745
0.5	448
0.6	542
0.7	652
0.75	687
0.8	719
0.9	809

SLOPE = 55.9 mV

REFERENCE SOLUTIONS

E_{K^+}	K^+ (ppm)
0.1	202
0.2	397
0.3	584
0.4	774
0.5	480
0.6	559
0.7	667
0.8	—
0.9	849
1.0	935

SLOPE = 59.4 mV

0.8 E_{K^+} was skipped, see page 52 for explaination.

12/30/91 1315 TD

Remeasured 0.05 N NaCl/KCl solutions for K^+ (original measurements were recorded on pages 53 and 58)

E_{K^+}	K^+(ppm)
0.01	9.18
0.02	24.3
0.03	44.5
0.05	84.5
0.075	141
0.09	175
0.1	207
0.13	256
0.15	297
0.18	346
0.2	382
0.24	457
0.3	556
0.35	644
0.4	752
0.5	85.2
0.6	105
0.7	118
0.75	126
0.8	144
0.9	149

SLOPE = TD 12/30/91 56 56.3

REFERENCE SOLUTIONS

E_{K^+}	K^+(ppm)
0.1	184
0.2	370
0.3	559
0.4	747
0.5	901
0.6	101
0.7	115
0.8	133
0.9	147
1.0	165

SLOPE = 55.5 mV

12/30/91 T.G. 1000

Preparation of $NaHCO_3/KHCO_3$ (0.05N) Solution

Objective: To prepare various $NaHCO_3/KHCO_3$ solutions, each of which having a total normality of 0.05N.

Method: Serial dilution from 0.5 N solutions (pg. 170)

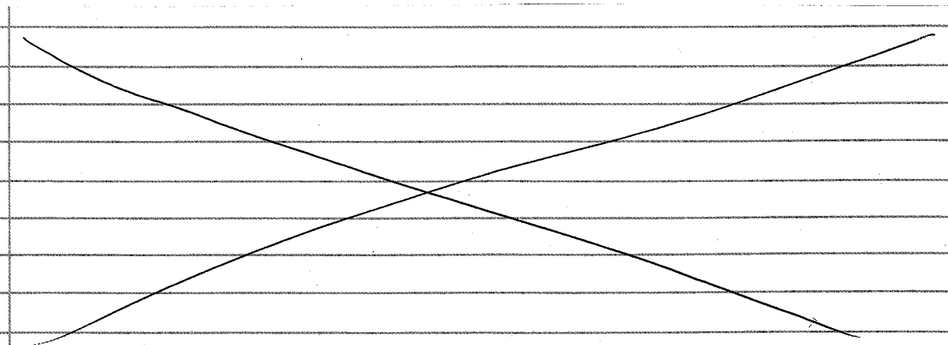
Equipment: 10-1L- polypropylene Nalgene bottles
100 ml pipet and bulb
1000-ml volumetric flasks
eye dropper

Reagents: previously made 0.5N $NaHCO_3/KHCO_3$ solutions
> 10 L deionized H_2O (@ > 17 MΩ resistivity)

Procedure:

2. Make up 1000 ml of 0.05N $NaHCO_3$-$KHCO_3$ solution by pipetting 100 ml of the 0.5 N solution and diluting it with ultrapure water in a 1000 ml volumetric flask. Make sure to initially rinse the pipet with a few ml of the 0.5 N solution.

Transfer the 0.05 N solution into a 1000 ml polypropylene bottle. Label the bottle (e.g., $NaHCO_3/KHCO_3$*0.05N*0.1X_K, plus Date and Initial).



12/31/91 0855 TD

Remeasured 0.005N NaCl/KCl solutions for K^+ (original measurements were recorded on pages 53 and 59).

E_{K^+}	K^+ (ppm)
0.01	.901
0.02	2.12
0.03	4.25
0.05	8.60
0.075	14.3
0.09	17.3
0.1	19.2
0.13	27.7
0.15	30.9
0.18	35.7
0.2	42.8
0.24	47.2
0.3	65.9
0.35	65.3
0.4	76.9
0.5	89.7
0.6	107
0.7	126
0.75	130
0.8	141
0.9	154

SLOPE = 57.1 mV

Reference Solutions

E_{K^+}	K^+ (ppm)
0.2	39.1
0.3	57.6
0.4	75.5
0.5	95.5
0.6	115
0.7	~~219~~ 119 TD 12/31/91
0.8	154
0.9	172
1.0	182

SLOPE = 57.3

The 0.1 E_{K^+} solutions was not measured. See page 53.

12/31/91 1045 TD

K^+ concentration measurements for the $KNaNO_3$ *.05N reference solutions were taken and recorded in Table 1 on page 151

SLOPE = 55.0 mV

1230 TD

Na^+ concentration measurements for the $CaNaNO_3$ *.05N reference solutions were taken and recorded in Table 1 on ~~154~~ TD original page 154

SLOPE = 55.2 mV

1319 TD

Na^+ concentration measurements for the $KNaNO_3$*.05N reference Solutions were taken and recorded in Table 1 on page 151

SLOPE = 55.2 mV

12/31/91 1430 T.G.

Preparation of CDV -- $NaHCO_3/KHCO_3$ (0.05N) Mixtures for Isotherm Experiment @ 25°C

Objective - to prepare 0.05N $NaHCO_3/KHCO_3$ -- CDV ~~Solutions~~ TG-mixtures by adding various amounts of zeolite to specific volumes of the 0.05N solutions; the zeolite is the CDV * 100/200 * UC * WH * HL * CPT * Naf and the solution (0.05N) is 0.05N $NaHCO_3/KHCO_3$ found on pg 185

Method - Weigh the CDV * ... * Naf in a bottle slightly larger than the amount of 0.05N $NaHCO_3/KHCO_3$ solution to be added, add the solution, and let the sample reach equilibrium @ 25°C

cont.

Equipment –
Mettler AE240, balance accurate to 0.001g (101237)
Spatulas
7.24.94 TG 21-30ml PP bottles
Versa Bath S, model 236

Reagents – 0.1-1.0 X_K ($NaHCO_3/KHCO_3$ @ 0.05N) see pg 185
CDV * 100/200 * dc * WA * HC * CPT * Naf

Procedure – 1.) Acid clean and dry 21-30ml PP bottles

2.) Label the bottles as :
$KNaHCO_3$ * 0.05
1

3.) For each sample, weigh the empty bottle and lid and record the weight

4.) Remove the cap and tare the balance. Add the appropriate 7.6 7-24-94 amount of CDV * ... * Naf (found in column 3 of Table I) to the empty bottle. Record the weight in the same table.

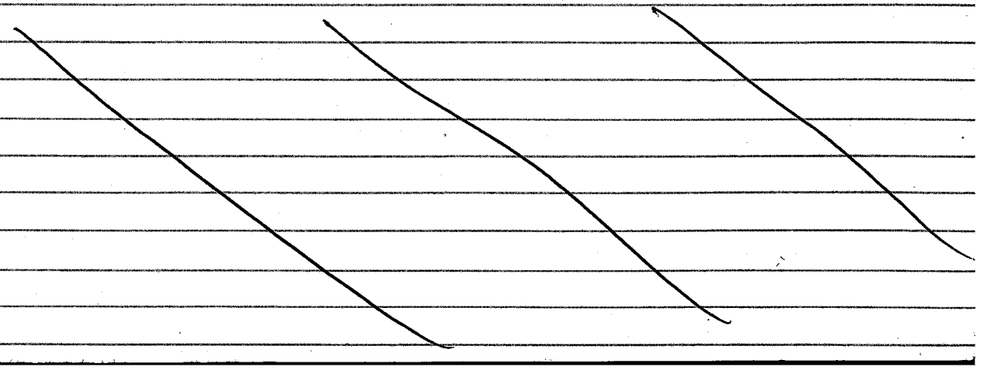
5.) Add 25 ml of the solution indicated in column 2 (Table I) to its respective sample The solution wt can be obtained by weighing the bottle w/ lid and solution. Record the weight in column 7 of the table. Subtract columns 5 and 6 from 7 to obtain the solution wt.

See the table on the next pg.

Table I

cont. $NaHCO_3/KHCO_3$ (0.05N) ISOTHERM EXT @ 25°C

Sample Label $KNaHCO_3$* 0.05*	$E_{i,K}$ used	Wt. Zeol. to use (gm)	Vol. Soln. Used (ml)	Wt. Zeol. Used (gm)	Wt. Bottle Empty (gm) w/Lid	Wt. Bottle + Soln. (gm) w/Lid
1	.1	.2592	25	.2591	7.9799	~~33.2330~~ 33.2349 TG 7-24-94
2	.1	.1536	25	.1536	7.9856	33.1445
3	.1	.1008	25	.1008	7.9449	33.0492
4	.2	.1694	25	.1693	7.9066	33.1028
5	.3	.2160	25	.2160	7.9265	33.1565
6	.3	.1890	25	.1890	8.0326	33.2401
7	.3	.1759	25	.1759	8.0022	33.2046
8	.3	.1399	25	.1399	8.0370	33.2051
9	.4	.1972	25	.1973	7.9901	33.2103
10	.4	.1690	25	.1691	8.0069	33.1912
11	.4	.1496	25	.1497	7.9953	33.1638
12	.4	.1152	25	.1152	7.9249	53.0523
13	.5	.1397	25	.1397	7.9621	83.1335
14	.6	.1694	25	.1695	7.9391	33.1200
15	.7	.1897	25	.1897	8.0163	33.2133
16	.8	.1931	25	.1931	7.9713	33.1771
17	.9	.1879	25	.1879	7.8936	33.1119
18	1.0	.1838	25	.1838	8.0233	33.2278
19	1.0	.1516	25	.1516	7.9503	33.1248
20	1.0	.1200	25	.1199	8.0587	33.2008
21	1.0	.0588	25	.0588	7.9414	33.0216



1/2/92 0900 T.C.

Remeasured all $NaNO_3/CaNO_3$ (0.05N) solutions from the 25°C experiment.

Purpose: to check the reproducibility of earlier Na^+/Ca^{2+} measurements.

0.05N $NaNO_3/Ca(NO_3)_2$ (@ 25°C)

Sample Lable $CaNaNO_3$ * 0.05	Na^+ ions actual (ppm)	Ca^{2+} ions actual (ppm)
1	111	21.2
2	109	34.7
3	106	56.8
4	103	71.2
5	98.8	94.5
6	93.9	134
7	88.5	177
8	81.5	215
9	76.7	253
10	66.9	321
11	57.0	389
12	51.6	* 495
13	45.4	* 453
14	40.3	* 491
15	33.5	537
16	28.2	559
17	22.0	586
18	15.9	636
19	10.7	664
20	7.90	662
21	5.04	687
slope	58.7 mV	28.8 mV

1/15/92

1/2/92 1300 T.C.

Remeasured all $NaNO_3/CaNO_3$ (0.05N) solutions from the 25°C experiment

Purpose: to check the reproducibility of earlier Na^+/K^+ measurements. 1/15/92

0.05N $NaNO_3/CaNO_3$ (@ 25°C)

Sample Label $CaNaNO_3$ * 0.05	Na^+ ions actual (ppm)	K^+ ions actual (ppm)
1	110	11.3
2	110	22.8
3	109	44.5
4	108	80.1
5	105	130
6	103	159
7	102	182
8	99.4	235
9	92.1	268
10	91.9	316
11	88.4	353
12	83.1	417
13	77.9	509
14	72.1	602
15	64.9	681
16	55.9	98.9
17	44.3	117
18	33.2	141
19	27.7	147
20	22.0	158
21	11.2	175
slope	58.7 mV	57.3 mV

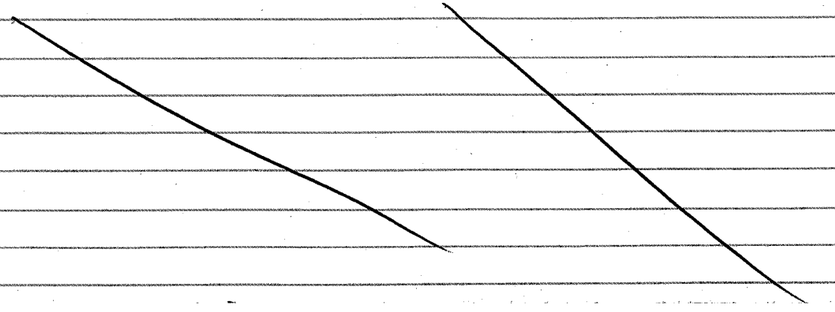
1/8/92 TD 1400 T.G.

Acid wash @ least 144 15 to 30 ml PP bottles, rinse each on thoroughly, and place in oven until completely dry. These bottles are to be used for the Reverse Isotherm experiment and the analyses of the 0.05 $NaKHCO_3$ solutions.

1/9/92 TD 0900 T.G.

To analyze the Reverse Isotherm solutions, make the necessary dilution using 10-ml volumetric flasks. Eppendorf pipets (0.5 and 1.0 ml) were used when required. To make the dilution, take a clean, acid-washed 10-ml volumetric (which has no water in it) and pipet the necessary volume. Rinse the pipet tip into the volumetric w/o 9-24-94 T.G. overflowing the volumetric. Fill to line. Transfer the solution to a clean acid-washed bottle. Label it eg.:

Rev. Iso. 25°C
E_{K^+}: 0.1
0.5

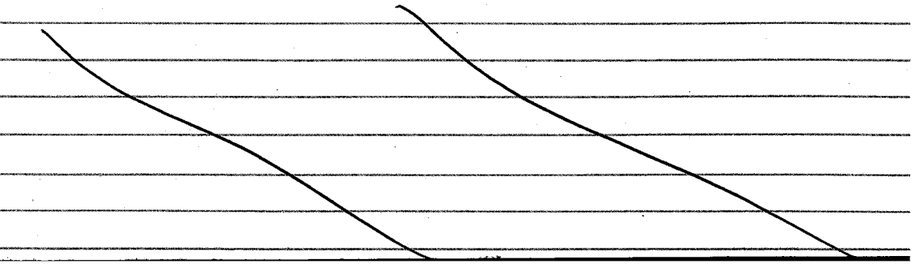


1/9/92 TD TD 0800

Remeasured the $NaCl/CaCl_2$ .005 N solutions for Na^+ (original measurements on page 63).

E_{Ca}	Na^+ (ppm)
.01	112
.03	111
.05	109
.08	107
.1	104
.13	98.9
.17	94.1
.2	~~89.2~~ TD 1/9/92 91.3
.23	89.4
.27	84.5
.3	82.4
.35	75.5
.4	69.3
.45	62.6
.5	57.5
.6	45.6
.65	38.2
.7	33.1
.75	27.3
.8	20.9
.85	15.4
.9	10.7
.95	5.35

SLOPE = 56.3 mV



1/9/92 TD 0945

Remeasured The reference solutions from the NaCl/$CaCl_2$ isotherm experiment (@25°C) (original measurements on pages 54 & 55)

E_{Ca}	Na^+(ppm) .5N soln	Na^+(ppm) .05N soln	Na^+(ppm) .005N soln
0.1	521	106	—
0.2	922	944	—
0.3	816	826	82.5
0.4	703	713	69.0
0.5	585	597	57.2
0.6	477	480	44.7
0.7	359	375	33.1
0.8	239	239	21.5
0.9	122	124	19.1 *
SLOPE	59.3mV	59.3mV	56.8mV

1/10/92 TD TG. 1000

Na^+ Analyses of the NaCl/KCl & NaCl/$CaCl_2$ Reverse Isotherm Exp @ 25°

Objective - to accurately measure the Na^+ concentration for each solution prepared in the experiment.

Method - measurements were made using an Orion 970A Ion Selective Meter and Na^+ electrode.

Equipment - Orion 970A Ion Selective Meter (S/N 001606)
Ross Sodium Electrode w/ BNC Connector Orion Cat.# 208574-A02

Reagents - Orion 100 ppm Std. Solution
" 1000 ppm Std. Solution
" Ionic Strength Adjustor.

KCl/NaCl Solutions Na^+ Analyses cont.

Sample #	E_{K^+}	0.5N Na^+ ions (505)	0.05N Na^+ ions	0.005N Na^+ ions
1	.01	414	107	106
3	.03	385	87.4	88.5
5	.075	397	70.4	91.2
7	.1	305	65.0	86.0
9	.15	544	62.4	77.5
11	.2	445	55.9	67.1
13	.3	409	49.0	61.9
15	.4	353	T.O. 76.5	52.5
17	.6	580	51.4	54.1
19	.75		40.0	28.7
slope		58.6	58.4	58.9

These are the actual conc measured. Below are the corrected solution analyses.

Sample #	Dilution Factor 0.5	0.05	0.005	Corrected Na^+ Conc. 0.5	0.05	0.005
1	.5/10	1/10	1/1	10100	1070	106
3	.5/10	1/10	1	8280	874	88.5
5	.5/10	1/10	1	7700	704	91.2
7	.5/10	1/10	1	7940	650	86.0
9	.5/10	1/10	1	6100	624	77.5
11	1/10	1/10	1	5440	559	67.0
13	1/10	1/10	1	4450	490	61.9
15	.5/10	1/10	1	8180	T.O. 765	52.5
17	.5/10	1/10	1	7060	514	54.1 * See pg 179
19	1/10	1/10	1	5800	400	28.7

TG 7-24-94

$CaCl_2$/NaCl Solutions
Na^+ Analyses cont.

Sample #	0.5 Eca^{2+}	0.5 N Na^+ ions	0.05 N Na^+ ions	0.05 Eca^{2+}	0.005 Eca^{2+}	0.005 Na^+ ions
1	.02	462	93.3	.015	.01	101
3	.06	459	89.0	.05	.05	79.9
5	.1	392	65.5	.1	.1	70.9
7	.15	303	58.7	.2	.17	58.8
9	.25	267	49.2	.3	.23	73.0
11	.4	353	36.6	.5	.3	66.8
13	.6	232	65.2	.6	.4	57.2
15	.7	708	55.9	.7	.5	48.1
17	.8	682	51.9	.8	.85	33.5
19	.87	445	28.4	.9	.95	23.7
					7.24.94	
slope		58.6	59.2			58.9

Sample #	Dilution Factor 0.5	0.05	0.005	Corrected Na+ concentration 0.5	0.05	0.005
1	.5/10	1/10	1	9240	933	101
3	.5/10	1/10	1	8780	890	79.9
5	.5/10	1/10	1	7840	655	70.9
7	.5/10	1/10	1	6060	587	58.8
9	.5/10	1/10	1	5340	492	73.0
11	1/10	1/10	1	3530	366	66.8
13	1/10	1/10	1	2320	652	57.2
15	1/10	1/10	1	7080	559	48.1
17	1/10	1/10	1	6820	519	33.5
19	1/10	1/10	1	4450	284	23.7

1/10/97 T.D. T.G. 1600

Ca^{2+} Analyses of the $CaCl_2$/NaCl Reverse Isotherm Experiment @25°C

Reagents - Orion 0.1 M Calcium std.
5M KCl Ionic Strength Adjustor

Sample #	0.5 Eca^{2+}	0.05 Eca^{2+}	0.005 Eca^{2+}	0.5 Na^+ ions	0.05 Ca^{2+} ions	0.005 Ca^{2+} ions
1	.02	.015	.01	101	156	9.96
3	.06	.05	.05	141	202	26.8
5	.1	.1	.1	168	421	35.3
7	.15	.2	.17	237	471	45.8
9	.25	.3	.23	281	546	34.0
11	.4	.5	.3	732	692	39.6
13	.6	.6	.4	818	415	48.8
15	.7	.7	.5	388	496	56.3
17	.8	.8	.85	456	562	69.3
19	.87	.9	.95	608	777	79.0
			7.24.94			
slope				29.0	29.0	29.5

Sample #	Dilution Factor 0.5	0.05	0.005	Corrected Ca^{2+} concentration 0.5	0.05	0.005
1	.5/10	1	1	2020	156	9.96
3	.5/10	1	1	2820	202	26.8
5	.5/10	1	1	3360	421	35.3
7	.5/10	1	1	4740	471	45.8
9	.5/10	1	1	5620	546	34.0
11	1/10	1	1	7320	692	39.6
13	1/10	1	1	8180	415	48.8
15	1/10	1	1	3880	496	56.3
17	1/10	1	1	4560	562	69.3
19	1/10	1	1	6080	777	79.0

1/13/92 0800 TD

The $KNaHCO_3$ *.05 ~~N~~ TD solutions were removed from the constant temperature bath. Two samples were taken from each solution, One sample for Na^+ concentration analysis and one for K^+ Concentration analysis. The Na^+ ~~solutions~~ 1/13/92 TD samples had a dilution factor of 1/10 while the K^+ ~~solutions~~ samples TD had a dilution factor of 1. The samples were placed in acid washed and ~~dried~~ 1/13/92 TD dried 15-mL PP bottles labled

$KNaHCO_3$ *.05 ~~N~~ TD 1/13/92 or $KNaHCO_3$ *.05
10 Na^+ 7 K^+

and the appropriate ISA was added. Tim Griffin took the K^+ samples from the $KNaHCO_3$ *.05 solutions.

1/14/92 0800 T.G.

After all the necessary dilutions were made (see top of pg.), the Na^+ and the K^+ concentrations for the $NaHCO_3$ - $KHCO_3$ (0.05) Isotherm Experiment were measured. These measurements follow on ~~the next~~ T.G 1/14/92 pg. 199.

The K^+ solutions for the Reverse Isotherm Experiment were diluted on 1/9/92 (see pg 199 T.G.) and will also be analyzed today ~~with~~ T.G. since the K^+ electrode will be set up. These results ~~will~~ T.G. can be found on pg 200.

Cont.

$NaHCO_3$-$KHCO_3$ (0.05 N) Isotherm Experiment

$E_{i,K}$ used	Sample Dilution Factor		Diluted Sample Concentration (ppm)		Reference Dilution Factor		Diluted Reference Concentration (ppm)	
	Na+	K+	Na+	K+	Na+	K+	Na+	K+
.1	1/10	1	113	8.20 ~~165~~ T.G. 7-24-94	10/100	1	102	180
.1	1/10	1	111	20.9 ~~148~~ "	-	-	-	-
.1	1/10	1	108	43.1 ~~137~~ "	-	-	-	-
.2	1/10	1	106	81.5 ~~129~~ "	10/100	1	89.8	365
.3	1/10	1	103	131 ~~113~~ "	10/100	1 T.G. 7-24-94	78.1	545
.3	1/10	1	102	164 ~~93.8~~ "	-	-	-	-
.3	1/10	1	99.4	180 ~~717~~ T.G. 7-24-94	-	-	-	-
.3	1/10	1	96.9	232	-	-	-	-
.4	1/10	1	94.4	276	10/100	1 T.G. 7-24-94	66.2	717
.4	1/10	1	92.7	326	-	-	-	-
.4	1/10	1	88.6	367	-	-	-	-
.4	1/10	1	83.2	433	-	-	-	-
.5	1/10	1	77.9	536	1	1 T.G. 7-24-94	557	875
.6	1/10	1	72.6	622	1	10/100	451	115
.7	1/10	1	65.1	717	1	10/100	337	133
.8	1/10	1/10	55.0	93.8	1	10/100	267	150
.9	1/10	1/10	44.2	113	1	10/100	112	168
1.0	1/10	1/10	32.2	129	-	10/100	-	190
1.0	1/10	1/10	27.4	137	-	-	-	-
1.0	1/10	1/10	21.2	148	-	-	-	-
1.0	1/10	1/10	11.0	165	-	-	-	-

Electrode Slope: 57.9 : 57.0 57.3 : 57.0

Cont. K^+ Analyses of the KCl/NaCl Reverse Isotherms Exp. @ 25°C

Reagents- Corning 0.1 M K^+ Std.
Orion 5M NaCl Ionic Strength Adjustor

Sample #	T.C. ~~#.5~~ E_{K^+}	Diluted Sample Concentration 0.5N K^+ ions	0.05N K^+ ions	0.005 K^+ ions
1	0.01	239	128	13.3
3	0.03	511	466	39.2
5	0.075	620	760	38.0
7	0.1	777	851	45.2
9	0.15	976	935	58.8
11	0.2	1020	1000	77.4
13	0.3	1200	1120	82.4
15	0.4	270 ~~415~~ TC 924.94	588	105
17	0.6	415	1010	101 * See pg 179
19	0.75	949	1280	146
electrode slope		57.3	57.3	57.8

Sample #	Dilution Factor 0.5	0.05	0.005	Corrected K^+ Concentration 0.5	0.05	0.005
1	15/10	1	1	2390	128	13.3
3	15/10	1	1	5110	466	39.2
5	15/10	1	1	6200	760	38.0
7	15/10	1	1	7770	851	45.2
9	15/10	1	1	9760	935	58.8
11	1/10	1	1	10200	1000	77.4
13	1/10	1	1	12000	1120	82.4
15	.5/10	1	1	5400	588	105
17	.5/10	1	1	8300	1010	101
19	1/10	1	1	9490	1280	146

1/18/92 T.C. 1000

Preparation of $NaNO_3$-$CaCl_2$ Solutions (0.5N)

Objective- To prepare various $NaNO_3$/$CaCl_2$ solutions, all of which have a total normality of 0.5.

Method- Weigh various amount of both salts and dilute to 250 ml in a volumetric flask.

Equipment- Mettler AE240, balance accurate to .001g (serial# 1012337)
weighing bottle(s)
metal spatula(s)
150 ml beaker(s)
10-250 ml volumetric flask(s)
eye dropper
corning stirrer & magnets & magnetic rod.
10-250 ml Nalgene polypropylene bottle

Reagents- Calcium Chloride Dihydrate Lot # 4160 KENN
Sodium Nitrate Lot # 7808 KDJE
Ultrapure Water (>17 MΩ resistivity)

Procedure - **Preparation of $NaNO_3$-$CaCl_2$ Solutions**

Written by: R. T. Pabalan **Date:** January 16, 1992
Revision No.: 0 **Date Revised:**

1. Prepare 250 ml of $NaNO_3$-$CaCl_2$ aqueous mixture with a total normality of 0.5 N and a fixed Na/Ca ratio by taring reagent grade $NaNO_3$ and $CaCl_2 \cdot 4H_2O$ in the amounts given in Table 1 onto a clean (acid-washed) 250 ml beaker. Keep the beaker covered with parafilm between additions of reagents to minimize adsorption of water from the atmosphere. Add about 100 ml ultrapure water (>17 megaohm resistivity) to the beaker, add a stir bar and dissolve the salts completely on a stir plate (add more water if necessary). Retrieve the stir bar with a magnetic rod, then carefully rinse the rod and stir bar with ultrapure water, making sure to catch all the washings in the beaker.

Cat.

Decant the solution into a clean 250 ml volumetric flask (with the help of a glass rod); rinse the beaker (and glass rod) with ultrapure water several times, carefully transferring all washings into the volumetric flask. Then fill the flask to about 2-3 inches below the mark with ultrapure water; swirl or shake the flask. Let stand for a few seconds, then add water dropwise up to the mark; remix.

Transfer the solution into a clean 250 ml polypropylene bottle. Label the bottle (e.g., $NaNO_3/CaCl_2$*0.5N*0.1X_K, plus Date and Initial).

TABLE I

X_K (0.5 N) ($NaNO_3$-$CaCl_2$ soln.)	Wt. $NaNO_3$ needed for 250 ml	Wt. $CaCl_2.2H_2O$ needed for 250 ml	Wt. $NaNO_3$ used	Wt. $CaCl_2.2H_2O$ used
0.1	9.5618	0.91885	9.5619	.9189
0.2	8.4994	1.8377	8.4995	1.8378
0.3	7.4370	2.7566	7.4369	2.7566
0.4	6.3746	3.6754	~~3.6753~~ 6.3744 TG 2/4/94	3.6753
0.5	5.3121	4.5943	5.3120	4.5944
0.6	4.2497	5.5131	4.2496	5.5131
0.7	3.1873	6.4320	3.1873	6.4319
0.8	2.1248	7.3508	2.1248	7.3505
0.9	1.0624	8.2697	1.0623	8.2694
1	0.0000	9.1885	0.0000	9.1884

1/19/92 TG. 1000

Preparation of $NaNO_3/CaCl_2$ (0.05N) Solutions

2. Make up 1000 ml of 0.05N $NaNO_3$-$CaCl_2$ solution by pipetting 100 ml of the 0.5 N solution and diluting it with ultrapure water in a 1000 ml volumetric flask. Make sure to initially rinse the pipet with a few ml of the 0.5 N solution.

Transfer the 0.05 N solution into a 1000 ml polypropylene bottle. Label the bottle (e.g., $NaNO_3/CaCl_2$*0.05N*0.1X_K, plus Date and Initial).

1/28/92 TD

Preparation of CDV*...*Naf - $NaNO_3$ - $CaCl_2$ (0.05N) samples at 25°C

OBJECTIVE: to prepare samples consisting of CDV*...*Naf powder in 0.05N $NaNO_3$-$CaCl_2$ solutions for isotherm experiments at 25°C.

METHOD: add CDV*...*Naf powder to 0.05N NO_3-Cl solutions of varying concentrations of Na and Ca and allow the sample to equilibrate at 25°C

EQUIPMENT:

a) Mettler AE240 balance accurate to 0.001g (SN 101237)
b) 30ml PP bottles
c) 60ml PP bottles
d) 25ml pipettes
e) 50ml pipettes
f) spatulas
g) VersaBath S, Model 236 constant temperature bath

REAGENTS

a) 0.1-1.0 X_K 0.05N $NaNO_3$-$CaCl_2$ solutions prepared previously (p. 202)
b) CDV*...*Naf powder

PROCEDURE

1) ACID clean and dry PP bottles and pipettes
2) label the bottles
$NaNO_3/CaCl_2$*.05*(1-20) as shown in the table
3) For each sample, weigh the bottle and tare the balance. Add the ~~CDV*Na~~ 1/28/92 TD CDV*...*Naf powder to the weight shown in Table 1 (p. 204). Record the actual weight in Table 1.

~~TD~~ 1/21/92 Preparation of 0.05N $NaNO_3/CaCl_2$ isotherm solutions (cont)

Procedure

4) Remove bottle from the scale. Add the volume of the appropriate E_i Ca^{2+} solution (both shown in Table 1. Weigh bottle with solution and record the weight in Table 1.

5) Place bottles in a constant temperature bath at 25°C and 60 rpm.

TABLE 1: $NaNO_3/CaCl_2$ .05 N ISOTHERM @ 25°C

	E_i Ca^{2+} used	wt seed to use (gm)	Vol. Soln. Used (gm)	Dilution Factor Na^+	Dilution Factor Ca^{2+}	wt seed Used (gm)	wt soln Used (gm)	Expected Sample Diluted Na^+	Expected Sample Diluted Ca^{2+}	Reference Dil. Fact. Na	Reference Dil. Fact. Ca^{2+}	Reference Expected Diluted Na^+	Reference Expected Diluted Ca^{2+}
1	.1	.2448	25	1/40	1	.2450	24.9099	113	15	1/10	1	103	100
2	.1	.1493	25	1/40	1	.1495	24.8396	112	30	—	—	—	—
3	.2	.2880	25	1/10	1	.2883	24.9743	109	50		1	920	200
4	.2	.2033	25	1/40	1	.2035	24.9272	106	80		—	—	—
5	.2	.1557	25	1/10	1	.1560	24.9219	104	100		—	—	—
6	.3	.2057	25	1/10	1	.2060	25.0337	98	150		1	805	301
7	.4	.2560	25	1/10	1	.2558	25.0098	92	200		1	690	401
8	.1	.1800	25	1/10	1	.1802	25.0133	86	251	—	—	—	—
9	.5	.2304	25	1/10	1	.2304	24.9764	81	301		1	575	501
10	.6	.2174	25	1/10	1	.2175	24.8513	69	401	1		460	601
11	.7	.2021	25	1/10	1	.2021	24.9594	58	501			345	701
12	.7	.1465	25	1/10	1	.1466	24.9428	52	551	—		—	—
13	.8	.1858	25	1/10	1/10	.1858	24.9857	46	601	1		230	802
14	.9	.2323	25	1/10	1/10	.2325	24.9702	40	651			115	902
15	1.0	.2541	25	1/10	1/10	.2538	24.9755	35	701	—		—	1002
16	1.0	.2000	25	1/10	1/10	.1998	24.9209	29	752	—		—	—
17	1.0	.1516	25	1/10	1/10	.1519	25.0158	23	802	—	—	—	—
18	1.0	.2134	50	1/10	1/10	.2132	49.9224 50.0120	17	852	—	—	—	—
19	1.0	.1355	50	1/10	1/10	.1356	49.8304	12	902	—	—	—	—
20	1.0	.0640	50	1/10	1/10	.0637	49.7332	6	952	—	—	—	—

1/29/92 TD 11:30

Samples of the $NaNO_3/CaCl_2$ (0.05N) isotherm experiment solutions were taken and diluted according to Table 1 on pg. 204. The appropriate Ionic Strength adjuster, either Na^+ or Ca^{2+}, was added. The dilutions were placed in acid cleaned and dry 15ml PP bottles labled $NaNO_3/CaCl_2$ *.05 (1 Na^+) or $NaNO_3/CaCl_2$ *.05 (10 Ca^{2+}) according to which ISA was added.

1/30/92 1030 TD

The Solutions used in the $NaNO_3 - Ca(NO_3)_2$ .05N isotherm experiments were resampled for Na and Ca measurements. The dilution factor for ALL samples is 1/10. The appropriate ISA was added (Either Na^+ or Ca^{2+}) and the dilutions were placed in acid cleaned and dried 15ml PP bottles labled $NaCaNO_3$ *.05* II (1 Na^+) or $NaCaNO_3$ *.05 * II (12 Ca^{2+}) depending on which ISA was added.

2/3/92 0800 T.O.

Over the last two weekends, 1/24/92 → 1/26/92 to 1/31/92 → 2/2/92, all the prepared CDV solutions were reanalyzed to ~~ensure~~ T.O. 7-24-92 ensure that the previous analyses ~~were~~ T.O. 7-24-92 were correct.

On the following pages are the remeasured analyses of these solutions: Rev. Iso. Exp., .05 $CaNaNO_3$, .05 $KNaHCO_3$, .05 $KNaNO_3$, .05 $NaNO_3/CaCl_2$, and all the corresponding references.

cont. Remeasured T.W.

Reverse Isotherm Exp. (NaCl/kCl)

Sample #	0.5 NaKCl		0.05 NaKCl		0.005 NaKCl	
	Na^+	K^+	Na^+	K^+	Na^+	K^+
1	510	237	107	134	109	12.8
~~3~~ 2	421	510	86.5	470	94.9	38.2
~~5~~ 3	389	620	69.3	766	95.3	37.3
~~7~~ 4	398	770	64.8	844	90.0	45.2
9	303	966	60.4	935	81.4	57.7
11	548	1010	53.4	1020	68.5	75.6
13	447	1180	46.6	1160	61.6	87.1
15	412	273	77.1	604	50.8	104
17	342	408	50.7	1030	52.9	100
19	565	950	39.4	1350	27.9	143
slope:	58.5	56.8	56.2	57.7	56.2	57.1
date:	1/25/92	2/2/92	2/1/92	2/2/92	2/1/92	2/2/92

Reverse Isotherm Exp. (NaCl/$CaCl_2$)

Sample #	0.5 NaCl/$CaCl_2$		0.05 NaCl/$CaCl_2$		0.005 NaCl/$CaCl_2$	
	Na^+	Ca^{2+}	Na^+	Ca^{2+}	Na^+	Ca^{2+}
1	465	103	92.7	152/158	108	9.31
3	441	143	88.6	196/203	85.1	26.9
5	393	170	65.0	406/423	73.3	35.5
7	296	236	57.0	450/469	59.5	47.0
9	263	279	48.7	525/548	75.5	34.2
11	355	721	35.3	663/690	68.3	40.2
13	226	797	63.5	391/408	57.4	49.9
15	712	375	55.0	468/487	47.3	51.1
17	693	436	49.5	532/553	32.9	71.2
19	451	600	27.0	735/765	23.2	80.1
slope	58.1	28.0	56.2	28.1/28.5	56.2	28.1
date	1/25/92	1/26/92	2/1/92	1/26/92	2/1/92	1/26/92
temp		63.3°F		63.4°F		63.4°F

T.W. 2/2/92

cont.

Remeasured References (Nitrates & Bicarbonates)

Sample #	0.05 NaCaNO3		0.05 NaKNO3		0.05 NaKHCO3	
	Na^+	Ca^{2+}	Na^+	K^+	Na^+	K^+
0.1	100/99.1	79.7	97.5	176/185	97.6/98.3	181
0.2	873/897	151	920	361/359	893	357
0.3		221	802	549/548	768	537
0.4	659/692	285	679	688/711	667 T.W. 7-24-94	721
0.5	552/571	349	573	883/903	580/563	881
0.6	441/460	411	471	/114	453/458	117
0.7	329/347	466	351	/134	343/341	136
0.8	230	530	230	/152	264/267	153
0.9	113	591	118	/171	117/115	171
1.0	—	647	—	/193	—	192
slope	58.2/59.1	28.0	59.1	/57.1	59.1/59.1	58.0
temp		63.2°F		19.5°c		19.5°c
date.	1/25/92	1/26/92	1/25/92	2/2/92	1/25/92	2/2/92

0.05 $NaCaNO_3$ Ref.

Ca^{2+} ions

Sample	
~~0.0~~	~~T.W. 7-24-94~~
~~0.1~~	~~156~~ 80.7
~~0.2~~	~~228~~ 156
~~0.3~~	~~295~~ 228
0.4	295
0.5	361
0.6	425
0.7	489
0.8	551
0.9	612
1.0	671
slope	28.5 on 2/2/92

cont. Remeasured Nitrates

Sample #	0.05 NaCaNO3 Na+	0.05 NaCaNO3 Ca2+	0.05 NaKNO3 Na+	0.05 NaKNO3 K+	
1	111	20.2	109	T.G. 11.5 ~~8.87~~	TO 7-24-94
2	108	33.1	111	22.1	
3	105	53.9	109	44.2	
4	103	67.6	108	82.5	
5	99.8	88.6/88.7	105	135	
6	94.6	125/126	104	166	
7	88.8	163/167	103	186	
8	81.4	195/199	99.2	242	
9	76.4	229/234	93.7	268	
10	67.1	290/294	92.4	321	
11	55.8	/357//362	88.7	348	
12	51.9	/460//465	83.8	409	
13	45.3	/421//426	77.4	519	
14	/40.2	/452//460	72.4	603	
15	/33.2	/491//498	65.6	700?/780//788	
16	/27.6	/510//516	55.8	T.G. ~~85.9~~ 94.9	TO 7-24-94
17	/21.6	/523//530	44.4	113	
18	/15.7	/580//588	33.1	133	
19	/10.6	/593//603	27.8	141	
20	/7.96	/613//623	22.2	152	
21	5.05	/628//641	11.5	174	
slope	57.1/58.6	28.0/28.2//28.3	59.0	T.G. 2/9/92 56.8/57.5 ~~[illegible]~~	TO 7-24-94
temp		63.4°F		75.0/75.4	
date	1/25/91	1/26/92	1/25/92	2/9/92	

cont. Remeasured Bi Carbonates

Sample #	0.05 KNaHCO3 Na+	0.05 KNaHCO3 K+	
1	111	8.87	
2	108	22.0	
3	108	43.2	
4	107	80.0	
5	103	133	
6	102	166	
7	101	181	
8	96.3	233	
9	95.8	275//283	
10	92.1	318//327	
11	89.6	357//365	
12	84.8	//431	
13	79.0	//535	
14	73.0	//619	
15	65.8	//741	
16	56.1	/92.3	
17	44.6	/110	
18	32.4	/126	
19	27.5	/136	
20	21.6	/143	
21	10.8	/155	
slope	59.0	57.0/56.7//58.0	
temp.		69°F/69.9//70.9°F	
date	1/25/92	2/8/92	

cont.

	0.05N $NaNO_3/CaCl_2$ CDV Solutions			0.05N $NaNO_3/CaCl_2$ References	
Sample #	Na^+	Ca^{2+}	Sample #	Na^+	Ca^{2+}
1	115	19.8	0.1	103	85.6
2	113	33.0	0.2	909	167
3	110	50.7	0.3	799	248
4	108	73.4	0.4	686	334
5	106	89.8	0.5	573	421
6	99.0	136	0.6	460	518
7	93.9	182	0.7	345	615
8	92.4	216	0.8	232	740
9	82.4	263	0.9	116	1000
10	70.4	343	1.0	—	91.1
11	57.7	441			
12	52.2	480	slope	58.5	28.8
13	45.8	60.0/60.9	temp		
14	?31.2/31.8	? /73.2	date	2/8/92	2/9/92
15	43.0/42.9	/62.8			
16	37.4/38.1	/67.3			
17	32.0/32.1	/71.4			
18	28.3/28.8	/76.2			
19	21.0/21.4	/80.3			
20	17.0/16.3	/82.5			
T.G. ~~21~~ 7-24-94 TG					
slope	60.6/59.0	28.6/28.4			
temp		19.0°C			
date	2/1/92	2/2/92			

T.G. 2/9/92 1915 T.G.

The $NaNO_3/Ca(NO_3)_2$ solutions that Todd Dietrich prepared on 1/30/92 were analyzed to compared with earlier dilutions of the same solutions

~~0.05 Na~~ TG 7-24-94

$NaNO_3/Ca(NO_3)_2$ * .05 * II

signifies that these solutions were the 2nd samples taken.

Sample #	Na^+	Ca^{2+}
1	114	2.00
2	110	3.66
3	108	5.70
4	105	8.57
5	103	11.1
6	97.6	15.7
7	90.2	20.6
8	84.9	25.8
9	80.1	30.6
10	69.7	40.4
11	61.4	49.8
12	51.6	55.0
13	48.3	60.4
14	/40.8	65.9
15	/34.9	70.8
16	/27.6	76.8
17	/21.5	83.8
18	/15.8	86.8
19	/10.6	92.1
20	/8.18	93.9
21	/5.47	95.4
slope	58.5/57.6	28.9
date	2/8/92	2/9/92
temp.	75°F	70.9°F

Cont. T.G.

Reanalyzed the $NaNO_3/CaCl_2$ solutions. ~~which~~ The first analyses are found on pg 210 and were measured last weekend.

$NaNO_3/CaCl_2$ * .05 N

Sample #	Na^+	Ca^{2+}
1	113	19.4
2	111	31.6
3	108	48.7
4	102	71.1
5	102	85.8
6	96.4	137
7	91.8	182
8	88.4	220
9	79.1	266
10	67.8	342
11	56.5	435
12	50.5	480
13	43.9	56.5
14	30.0	69.3
15	41.3	60.0
16	36.1	64.9
17	30.3	69.0
18	26.3	75.6
19	20.0	78.8
20	15.6	83.4
slope	57.6	28.7
temp.	74.5	71.9
date	2/8/92	2/9/92

031392 — RJP

Preparation of clinoptilolite, analcime and NBS standards to be sent to Texas Tech for chemical analysis. ~ 0.5 gms of clinoptilolite (various exchanged forms) and NBS 70A and 99A were transferred into glass vials. Labels on vials correspond to sample names given below:

RJP 3/13/92

K — ~~[illegible]~~ CDV*100/200*UC*WA*HL*CPT*Na/Hf2
L — CDV* *Na/Hf3
M — CDV* *Na/Hf4
N — CDV* . . . . . *Na/Hf
O — NBS 99A
P — CDV* . . . . . *Na/Hf
Q — CDV* . . . . . *Caf
R — CDV* . . . . . *Kf
S — ASH*450/635*UC*WA
T — NBS 70A

041392 T.G.

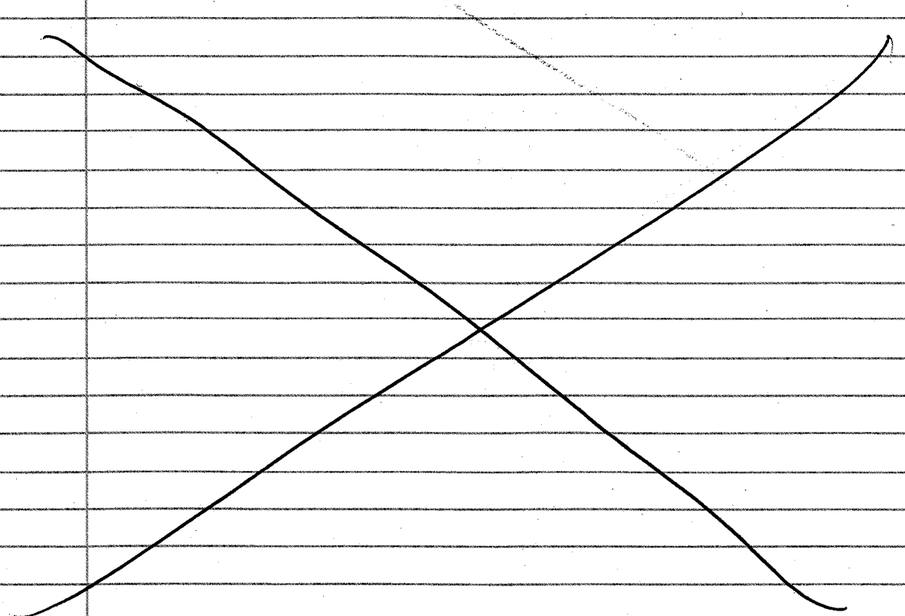
Prepared new solutions and standards to remeasure the T.G. 7.28.94 .05N $KNaHCO_3$ and the .05N $Ca(NO_3)_2/NaNO_3$ Reference solutions.

The purpose was to determine whether an error was made in earlier measurements of the solutions. The original preparation of these solutions can be found on pages 187-189 and 198-199.

041392 cont T.C.

Sample #	0.05 N $KNaHCO_3$ K^+ ppm	Ref #	0.05 $CaNaNO_3$ Ca^{2+}
14	65.0/65.2	0.1 X_{ca}	83.6
15	80.7/77.2	0.2 "	161
16	93.9/90.6	0.3 "	236
17	110/110	0.4 "	310
18	131/131	0.5 "	379
19	139/140	0.6 "	443
20	147/148	0.7 "	509
21	166/162	0.8 "	581
		0.9 "	649
slope	57.2	1.0 "	714

-slope



090492 RJP

Received results of chemical analysis from Texas Tech by FAX. Copy of results given below.

Zeolites

Sample	HS2 GRI K	HS3 GRI L	HSH GRI M	HF GRI N	99% GRI-O	HF GRI-P	CaF GRI-Q	KF GRI-R	ASH GRI-S	TOA GRI-T	Standards NHA	Standards AGMT
SiO2	67.98	67.53	69.41	69.12	64.23	67.61	66.44	65.97	53.77	67.56	69.45	73.55
TiO2	0.06	0.05	0.06	0.06	0.00	0.06	0.06	0.06	0.00	0.00	0.85	0.27
Al2O3	10.81	10.99	10.88	11.00	19.49	10.78	10.85	10.62	23.26	17.49	17.10	13.29
Fe2O3	0.37	0.35	0.40	0.40	0.04	0.36	0.41	0.40	0.01	0.05	6.23	1.92
MnO	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.10	0.04
MgO	0.17	0.17	0.18	0.17	0.05	0.17	0.22	0.16	0.03	0.06	3.08	0.29
CaO	0.03	0.03	0.04	0.04	2.03	0.03	3.71	0.03	0.03	0.14	6.05	1.17
Na2O	4.78	4.26	3.73	3.76	6.04	3.62	0.07	0.00	14.89	2.32	4.07	3.97
K2O	0.63	0.63	0.61	0.63	3.15	0.63	0.46	19.00	0.93	11.86	1.15	4.16
P2O5	0.03	0.02	0.01	0.02	0.02	0.01	0.02	0.03	0.01	0.01	0.17	0.05
TOTAL	84.85	84.03	85.33	85.18	97.03	83.26	84.24	87.28	92.03	99.69	99.26	98.70
Sr	8	8	9	9	425	8	89	4	5	64	540	103
Ba	126	128	129	127	3012	123	150	5	1	117	305	860
Zr	84	88	85	89	17	84	85	85	14	11	137	210
Y	7.9	7.8	8.1	7.5	1.5	7.8	7.7	8.1	0.0	1.0	16.3	25.2
Sc	1.2	1.2	1.2	1.2	0.2	1.1	1.2	1.2	0	0		
V	1	1	1	1	0	1	0	1	0	0		
Cr	4	4	3	3	2	2	2	3	41	1		
Ni	3	1	0	0	3	2	0	1	24	1		
Cu	5	1	0	0	6	1	2	1	2	5		
Zn	10	8	12	8	1	9	8	8	1	3		
Nb	18	16	17	17	0	18	18	17	1	2		
Rb	43	43	43	42	122	42	34	23	27	649		

5/6/92 T.G.

Solutions of $NaHCO_3/KHCO_3$ *.05N* II were prepared ~~in the same~~ T.G. following the procedures on pg 187-188. and pg 170-171. However, only the last 10 solutions were made and analyzed.

X_{K^+}	wt. $NaHCO_3$ needed for 250ml	wt $NaHCO_3$ used	wt. $KHCO_3$ needed for 250ml	wt $KHCO_3$ used
0.4	T.G ~~5.2504~~ 6.3005	6.3005	5.0057	5.0058
0.5	T.G ~~4.2003~~ 5.2504	5.2504	6.2571	6.2573
0.6	T.G ~~3.1502~~ 4.2003	4.2002	7.5086	7.5087
0.7	T.G ~~2.1002~~ 3.1502	3.1505	8.76	8.7602
0.8	T.G 2.1002	2.1002	10.011	10.0114
0.9	T.G 1.0501	1.0498	11.263	11.2632
1.0	0	0	12.514	12.5138

Sample #	E_{i,K^+}	wt. zeol. to use	wt. zeol. used.	wt. empty bottle w/ lid	25ml E_{i,K^+} soln + bottle and lid
12	.4	.1152	.1155	8.0251	33.1329
13	.5	.1397	.1397	7.8686	32.9908
14	.6	.1694	.1698	7.9592	33.1224
15	.7	.1897	.1895	7.9822	33.1773
16	.8	.1931	.1933	7.9786	33.1733
17	.9	.1879	.1878	8.0067	33.2028
18	1.0	.1898	.1835	7.9873	33.1800
19	1.0	.1516	.1516	7.9813	33.1400
20	1.0	.1200	.1202	7.9463	33.0716
21	1.0	.0588	.0587	7.9220	32.9886

5/28/92 T.G.

$NaHCO_3/KHCO_3$ * 0.05N * II solutions were measured using a Perkin Elmer 3100 Atomic Absorbtion Spectrometer at a wavelength, λ, of 766.5 nm. At this λ, the solutions had to be diluted to >2 ppm to fall in the linear range.

E_{i,K^+}	Dilution Factor	AA conc (ppm)	Actual conc
.4	1/1000	0.487	487
.5	1/1000	0.601	601
.6	1/1000	0.703	703
.7	1/1000	0.820	820
.8	1/1000	0.983	983
.9	1/1000	1.167	1167
1.0	1/1000	1.384	1384
1.0	1/1000	1.442	1442
1.0	1/1000	1.519	1519
1.0	1/1000	1.695	1695

In performing these measurements, a 1000 ppm K^+ Ref. Sol'n from Fisher Scientific and Lot #910846-18 was diluted to 2ppm to make the standard. The same T.G. 1000 ppm solution was diluted to 0.4 ppm as a check. The AA showed less than 5% error from expected.

T.G. 7-24-92

7/21/92 TD NaCl/KCl ION EXCHANGE AT 80°C

REMEASURED ALL Na^+ CONC. IN THE 0.5N, 0.05N, 0.005N SOLUTIONS THAT WERE SAMPLED EARLIER (p. 121, 112-3)

	0.5N, Na^+ conc. (ppm)		0.05N, Na^+ conc (ppm)		0.005N, Na^+ conc (ppm)
E_{K^+}	MEAS.	ACTUAL	MEAS.	ACTUAL	ACTUAL
0.01	595	11,900	144	1440	138
0.02	618/629	12200/12580	154	1540	124
0.03	697/712	13940/14240	121	1210	113
0.05	621	12420	117	1170	117
0.075	560	11200	107	1070	107
0.09	556/553	11120/11060	131	1310	109
0.1	569	11380	131	1310	127
0.13	516	10320	113	1130	98.5
0.15	580	11800	129	1290	96.1
0.18	565/598	11300/11960	95.0	950	92.1
0.2	470	9400	111	1110	
0.24	458	9160	119	1190	109
0.3	846	8460	82.9	829	88.3
0.35	842	8420	73.6	736	72.3
0.4	743	7430	85.4	854	65.1
0.5	584	5840	57.3	573	56.5
0.6	473	4730	55.6	556	50.0
0.7	322	3220	34.2	342	33.7
0.75	293	2930	30.6	306	28.1
0.8	243	2430	230	230	22.2
0.9	111	1110	131	131	11.5
	SLOPE = 58.2 mV		58.8 mV		58.8 mV

TD 7/21/92
~~IT APPEARED THAT~~ SOME OF THE SOLUTIONS WERE LESS THAN THE ORIGINAL 10mL (EVAPORATION?). THE E_{K^+} = 0.2, 0.005N SAMPLES[TD] WAS COMPLETELY GONE AND SO IT COULD NOT BE REMEASURED.

7/22/92 TD NaCl/KCl ION EXCHANGE AT 80°C

REMEASURED THE K^+ CONC. IN ALL SOL'N (0.5N, 0.05N, 0.005N) THAT WERE MEASURED EARLIER (p. 120, 122-113).

	0.5N, K^+ conc (ppm)		0.05N, K^+ conc. (ppm)		0.005N, K^+ conc (ppm)
E_{K^+}	MEAS.	ACTUAL	MEAS.	ACTUAL	MEAS/ACTUAL
0.01	26.0	260	28.7	28.7	2.90
0.02	56.7	567	61.1	61.1	5.11
0.03	95.9	959	90.6	90.6	12.4
0.05	133	1330	210	210	15.2
0.075	190	1900	291	291	23.6
0.09	234	2340	345	345	27.8
0.1	232	2320	278	278	32.9
0.13	* 320	3200	32.8	328	48.2
0.15	495	4950	39.9	399	49.0
0.18	434	4340	55.8	558	69.5
0.2	425	4250	660	610	91.7
0.24	523	5230	62.4	624	60.6
0.3	633	6330	23.9	239	823
0.35	699	6990	69.5	695	106
0.4	859	8590	157	1570	105
0.5	742	14840	129	1290	116
0.6	541	10820	152	1520	135
0.7	633	12,660	149	1490	170
0.75	709	14,180	182	1820	134
0.8	1350 ■	27,000	150	1500	153
0.9	754	15080	216	2160	175
	SLOPE = 54.2 mV, ⁴ = 54.0 mV		SLOPE = 54.6 mV		SLOPE = 54.6 mV

■ OUT OF CALIBRATION RANGE

MOST OF THE SOLUTIONS WERE LESS THAN THE 10ml INITIAL SAMPLE VOLUME

7/23/92 TD ION EXCHANGE AT 80°C

MEASURED THE K^+ CONC IN THE REDO SOLNS.

0.5N, K^+		0.005N K^+	
E_{K^+}	CONC (ppm)	E_{K^+}	CONC (ppm)
0.01	22.1	0.6	125
0.02	49.9	0.7	143
0.03	76.7	0.75	151
0.05	122	0.8	161
0.075	—	0.9	188
0.09	226		
0.1	248		
0.13	335		
0.15	359		
0.18	448		
0.2	563		
0.24	703		
0.30	722		
0.35	797		
0.4	931		
0.5	633		
0.6	710		
0.7	920		
0.75	848		
0.8	843		
0.9	940		

TD 7/23/92
~~MOST~~ ALL THE SOLN'S WERE LESS THAN 10mL; THEREFORE, NONE HAVE BEEN ADJUSTED FOR DILUTION. THE 0.075 SOLN WAS GONE, SO NO MEASUREMENT WAS MADE.

7/24/92 TD ION EXCHANGE AT 80°C

MEASURED THE Na^+ CONC. IN THE REDO SOLNS

0.5N, Na^+		0.005N, Na^+	
E_{K^+}	Na^+ CONC (ppm)	E_{K^+}	Na^+ CONC. (ppm)
0.01	723	0.6	42.0
0.02	569	0.7	35.8
0.03	578	0.75	36.7
0.05	602	0.8	30.2
0.075	534	0.9	11.2
0.09	644		
0.1	538		
0.13	580		
0.15	538		
0.18	565		
0.2	515		
0.24	448		
0.3	805		
0.35	786		
0.4	783		
0.5	555		
0.6	441		
0.7	369		
0.8	~~774~~ TD 220		
0.9	~~—~~ TD 7/24/92		
0.75	774		

7/24/92

MOST ALL THE SOLUTIONS WERE LESS THAN 10ml VOLUMES; THEREFORE, THE ~~CONCE~~ TD 7/24/92 DILUTION FACTORS WERE NOT USED TO ADJUST THE CONCENTRATIONS. THE ABOVE DATA ARE UNCORRECTED.

7/27/92 TD ION EXCHANGE ($NaCl/CaCl_2$) AT 80°C

Remeasured the Na^+ concentration in the 0.5N, 0.05N, 0.005N $NaCl/CaCl_2$ 80°C ion exchange experiment samples.

0.5N		0.05N		0.005N	
$E_{Ca^{2+}}$	Na^+ conc (ppm)	$E_{Ca^{2+}}$	Na^+ conc (ppm)	$E_{Ca^{2+}}$	Na^+ conc
0.02	611	0.015	115	0.01	133°
0.04	591	0.03	114	0.03	119
0.06	589	0.05	111	0.05	130°
0.08	634°	0.08	109	0.08	150°
0.1	554	0.1	107	0.1	118°
0.12	554/546	0.15	106	0.13	130°
0.15	516	0.2	97.7	0.17	113°
0.2	582	0.25	90.2	0.2	128°
0.25	479	0.3	85.5	0.23	106°
0.3	450	0.4	78.7	0.27	98.0
0.4	402	0.5	61.4	0.3	1260
0.5	334	0.55	55.2	0.35	89.7
0.6	547	0.6	50.3	0.4	88.00
0.65	285	0.65	46.4	0.45	76.4
0.7	215	0.7	39.5	0.5	—
0.75	169	0.75	31.3	0.6	544
0.8	147	0.8	24.2	0.65	46.3
0.83	128	0.85	185	0.7	39.9
0.87	193/183	0.9	117	0.75	32.5
0.9	139	0.95	58.0	0.8	25.6
0.98	91.1			0.85	—
0.95	63.4			0.9	12.4°
				0.95	6.76°

°= LESS THAN 10 mL SOLN (SAMPLES) NOT CORRECTED FOR DILUTION

7/30/92 $NaCl/CaCl_2$ ION EXCHANGE AT 80°C

Remeasured the Ca^{2+} concentration in the 0.5N, 0.05N, 0.005N $NaCl/CaCl_2$ 80C ion Exchange experiment samples

0.5N		0.05N			0.005N	
$E_{Ca^{2+}}$	Ca^{2+} conc (ppm)	$E_{Ca^{2+}}$	Ca^{2+} conc (ppm)		$E_{Ca^{2+}}$	Ca^{2+}
0.02	12.7	0.015	7.57	9.02	0.01	—
0.04	26.3	0.03	15.9	18.1	0.03	3.68
0.06	40.7	0.05	28.1	31.6	0.05	—
0.08	64.1	0.08	45.3	51.9	0.08	2.13
0.1	76.5	0.1	64.6	71.4	0.1	4.67
0.12	99.6/106	0.15	107	117	0.13	5.56
0.15	126	0.2	136	148	0.17	10.7
0.2	199/170	0.25	192	200	0.2	14.4
0.25	238	0.3	229	245	0.23	12.8
0.3	252	0.4	313	36.1	0.27	17.9
0.4	190	0.5	41.3	46.7	0.3	19.4
0.5	255	0.55	47.1 TD 7/30/92 52.7		0.35	25.2
0.6	301	0.6	53 58.3		0.4	29.8
0.65	366	0.65		62.3	0.45	36.3
0.7	368	0.7		68.1	0.5	41.2
0.75	790	0.75		81.1	0.6	54
0.8	722/434	0.8		83.5	0.65	61.5
0.83	436	0.85		95.5	0.7	74.5
0.87	445	0.9		92.5	0.75	75.1
0.9	398	0.95		88.4	0.8	111
0.93	434				0.85	104
0.95	505				0.9	92.5
					0.95	95.8

9/1/92 T.6.

Purpose: To prepare 0.5N NaCl-$^{90}SrCl_2$ solutions for the strontium ion-exchange experiments.

Reagents:
Sodium Chloride (A.C.S. Certified) Lot # 912763
Strontium Chloride (A.C.S. Certified) Lot # 884315
Several Liters of Nanopure H_2O.

Materials:
AE 240 Mettler Balance (Serial # 8 101237)
Beakers
Class A, 500-ml volumetric flasks
weighing spatulas
Corning stirrer with magnetic stir bars.
Wash bottle and eyedropper.

Procedure:

To prepare the 0.5 total normality solutions, various ratios of the above chloride reagents were mixed together. Following is a variation and the actual procedure that was used in the weighing of these ratios; however, this written procedure only replaces the first ~~two~~ sentences of the typed procedure. The rest of the procedure will be conducted as it ~~is~~ was typed by R. T. Pabalan.

Table 1 shows how the various weight ratios provide us w/ a total normality of 0.5. The weight ratios were deduced from the optimization of the volume to solution and the Teralite to solution values which were calculated in a spreadsheet of Lotus 123.

cont.

TABLE 1

0.5 N of SrCl2/NaCl Solutions

Wt.SrCL2 in 500 ml	Wt.NaCl in 500 ml	MW.SrCL g/mol	MW.NaCl g/mol	Tot.Vol (ml)	M(Sr) mol/L	M(Na) mol/L	TN(Na+Sr equiv/L
1.6664	13.88	266.62	58.44	500	0.0125	0.475017	0.500017
3.3327	13.15	266.62	58.44	500	0.025	0.450034	0.500033
6.6654	11.689	266.62	58.44	500	0.049999	0.400034	0.500033
9.9981	10.228	266.62	58.44	500	0.074999	0.350034	0.500032
13.331	8.7664	266.62	58.44	500	0.1	0.300014	0.500014
16.664	7.3054	266.62	58.44	500	0.125002	0.250014	0.500017
19.996	5.8443	266.62	58.44	500	0.149996	0.20001	0.500003
23.329	4.3832	266.62	58.44	500	0.174998	0.150007	0.500003
26.662	2.9221	266.62	58.44	500	0.2	0.100003	0.500003
29.994	1.4611	266.62	58.44	500	0.224994	0.050003	0.499992
33.327	0	266.62	58.44	500	0.249996	0	0.499992

To weigh out the above $SrCl_2 \cdot 6H_2O$ and NaCl amounts, obtain 2 40-ml beakers (acid-washed) and label one beaker Na and the other Sr and only use that labeled beaker which corresponds to the reagent in the weighing. See Table 2 to find ~~the Eq~~ $E_{Sr,i}$ (or Equivalent initial strontium). pg 228
In the blue squares are the same values seen above. These values should be referenced when $E_{Sr,i}$ is referred.

In making the $E_{Sr,.05}$ solution, the Na labeled 40-ml beaker was tared on the AE240 Mettler balance. Using a spatula, approximately 13.66 g NaCl was transfered into this beaker and ~~recorded the actual weight~~ the actual weight was recorded in Table 2. Obtain a Clean, acid-washed 500-ml beaker and place ~200 ml of DI water into it. Sufficiently

cont

rinse all of the NaCl reagent into this 500-ml beaker. With the 40-ml bottle empty (and well-rinsed), dry it and set it aside for the ~~0.1~~ $E_{Sr,0.1}$ solution preparation. Now obtain the Sr labeled 40-ml beaker and transfer 1.6664 g $SrCl_2 \cdot 3H_2O$ to the beaker. Record the actual weight, like that in Table 2, and then wash all of the $SrCl_2 \cdot 3H_2O$ reagent into the same 500-ml beaker as that which contains the NaCl reagent. With the Sr beaker well-rinsed (and all washings in the 500-ml beaker), dry it and set it aside. ~~contain~~ T.G. 7-24-94

T.G. 7-24-94 ~~T.G. In~~ The 500-ml should ~~be~~ T.G. both chloride reagents in their predetermined 0.05 $E_{Sr,i}$ ratio.

* One should be aware that at this point, the DI water volume in the 500 ml beaker should not exceed 350 ml since it too is rinsed with all of its washings going into a clean (acid-washed) 500-ml Volumetric flask. ~~With~~ T.G. 7-24-94 Place the less than 350 ml solution onto a stir plate and completely dissolve the salts as written in the second sentence of the following typed article.

~~Follow the following procedure preci~~ T.G. 7-24-94

For each $E_{Sr,i}$, precisely weigh each reagent into its own clean, acid washed 500-ml beaker ~~using~~ T.G. 7-24-94 while identically following all but the reagent weight additions in the procedure above.

Follow the following procedure precisely. ~~except~~ T.G. 7-24-94 However, the 1 b., 2 b., and 3 b. procedures will be performed after the Strontium spike has been made.

cont.

Procedure for Preparation of NaCl-$^{90}SrCl_2$ Solutions

WRITTEN BY: R.T. Pabalan RT DATE: September 30, 1992
REVISION NO.: 0 DATE REVISED:

use smaller beaker. T.G. Oct 1, 1992

1. Prepare 500 ml of NaCl-$SrCl_2$ aqueous mixture with a total normality of 0.5 N and a fixed Na/Sr ratio by taring reagent grade $SrCl_2 \cdot 6H_2O$ in the amount given in Table 1 onto a clean 500 ml beaker. Add about 300 ml deionized water to the beaker, add a stir bar and dissolve the salts completely on a stir plate (add more water if necessary). Retrieve the stir bar with a magnetic rod, then carefully rinse the rod and stir bar with deionized water. Decant the solution into a clean 500 ml volumetric flask (with the help of a glass rod); rinse the beaker (and glass rod) with deionized water several times, carefully transferring all washings into the volumetric flask. Then fill the flask to about 2-3 inches below the mark with deionized water; swirl or shake the flask. Let stand for a few seconds, then add water dropwise up to the mark; remix.

b.) For each solution prepared above:

On a Mettler AE240 balance, tare a 200 µg aliquot (record the actual weight) of a 0.5 µCi/g ^{90}Sr spike on a plastic boat. On a Mettler PM4600 balance, tare a pre-labeled (e.g., Na/Sr90*0.5N*0.1X_{Sr}, plus Date and Initial) 250-ml polypropylene bottle, and carefully transfer the ^{90}Sr aliquot into the bottle. Rinse the boat with the solution and transfer all washings into the bottle. Transfer a total of 200 g of the NaCl-$SrCl_2$ solution into the bottle (final solution weight=200.20 g). Mix well and measure the pH of the solution. Put a radioactive symbol sticker on the bottle.

Transfer the remaining solutions into clean 500 ml polypropylene bottles. Label the bottle (e.g., Na/Sr*0.5N*0.1X_{Sr}, plus Date and Initial). Measure the pH of each solution.

2. Make up 1000 ml of 0.05N NaCl-$SrCl_2$ solution (except the 0.05X_{Sr} solution) by pipetting 100 ml of the 0.5 N solution and diluting it with deionized water in a 1000 ml volumetric flask. Make sure to initially rinse the pipet with a few ml of the 0.5 N solution.

b.) For solutions with X_{Sr} equal to 0.1 to 0.9:

On a Mettler AE240 balance, tare a 250 µg aliquot (record the actual weight) of a 0.5 µCi/g ^{90}Sr spike on a plastic boat. On a Mettler PM4600 balance, tare a pre-labeled (e.g., Na/Sr90*0.05N*0.1X_{Sr}, plus Date and Initial) 250-ml polypropylene bottle, and carefully transfer the ^{90}Sr aliquot into the bottle. Rinse the boat with the solution and transfer all washings into the bottle. Transfer a total of 250 g of the NaCl-$SrCl_2$ solution into the bottle (final solution weight=250.25 g). Mix well and measure the pH of the solution. Put a radioactive symbol sticker on the bottle.

For the solution with X_{Sr} equal to 1.0:

Do as in the above paragraph, but use 500 ml of the solution and 500 µg of the

cont. on next pg

cont.

^{90}Sr spike (final solution weight = 500.50; in 500-ml polypropylene bottle).

Transfer the remaining 0.05 N solution into 1000 ml polypropylene bottles. Label the bottles (e.g., Na/Sr*0.05N*0.1X_{Sr}, plus Date and Initial). Measure the pH of each solution.

3. Make up 1000 ml of 0.005N NaCl-$SrCl_2$ solution by pipetting 100 ml of the 0.05 N solution and diluting it with deionized water in a 1000-ml volumetric flask. Make sure to initially rinse the pipet with a few ml of the 0.05 N solution.

b) For solutions with X_{Sr} equal to 0.1 to 0.9:

On a Mettler AE240 balance, tare a 500 µg aliquot (record the actual weight) of a 0.5 µCi/g ^{90}Sr spike on a plastic boat. On a Mettler PM4600 balance, tare a pre-labeled (e.g., Na/Sr90*0.005N*0.1X_{Sr}, plus Date and Initial) 500-ml polypropylene bottle, and carefully transfer the ^{90}Sr aliquot into the bottle. Rinse the boat with the solution and transfer all washings into the bottle. Transfer a total of 500 g of the NaCl-$SrCl_2$ solution into the bottle (final solution weight=500.50 g). Mix well and measure the pH of the solution. Put a radioactive symbol sticker on the bottle.

For the solution with X_{Sr} equal to 1.0:

Do as in the above paragraph, but use 1000 ml of the solution and 1000 µg of the ^{90}Sr spike (final solution weight = 1001.00; in 1000-ml polypropylene bottle).

Transfer the remaining 0.005 N solution into 500 ml polypropylene bottles. Label the bottle (e.g., Na/Sr*0.005N*0.1X_{Sr}, plus Date and Initial). Measure the pH of each solution.

TABLE 2

0.5 N NaCl-SrCl2 Solutions

ESr,i	SrCl2.6H2O in 500 ml	NaCl in 500 ml	Actual wt. SrCl2.6H2O	Actual wt. NaCl
0.05	1.6664	13.88	1.6669	13.8806
0.1	3.3327	13.15	3.3325	13.1502
0.2	6.6654	11.689	6.6658	11.6889
0.3	9.9981	10.228	9.998	10.2279
0.4	13.331	8.7664	13.3315	8.7667
0.5	16.664	7.3054	16.6645	7.3057
0.6	19.996	5.8443	19.9959	5.8442
0.7	23.329	4.3832	23.3291	4.3833
0.8	26.662	2.9221	26.6621	2.9225
0.9	29.994	1.4611	29.9943	1.4613
1	32.327	0	33.3273	0

9/5/92 T.G.

Procedures 2.) and 3.) on pages 228-229 were performed today. Every time a solution was made, the final and intermediate weights (amount of solution removed from bottle for further dilution) of the bottle were recorded using ~~[illegible]~~ 7-24-94 TG the PM4600 Mettler balance.

The weights give us a volumetric representation of the amount of solutions left. Thus, when the spikes are prepared, we know just how much solution is used.

TABLE 3

Solutions' Weight After Preparation of TN's

	500 ml		1000 ml			1000 ml	
	Wt(0.5 N)		Wt(0.05 TN)			Wt(0.005 N)	
E(Sr,i)	SBL	-100&R	BL	SBL	-100&R	BL	SBL
0.05	562.38	----	----	----	----	----	----
0.1	563.14	451.2	100.36	1098.6	977.9	101.67	1097.6
0.2	564	449.92	101.35	1100	985.7	101.27	1097.7
0.3	564.24	449.92	100.22	1098.9	975	100.48	1096.7
0.4	565.5	447.76	101.41	1100.2	969	101.14	1097.4
0.5	565.12	449.04	100.7	1099.7	976.9	100.34	1096.9
0.6	566.21	448.61	101.16	1100.2	967.2	101.05	1097.1
0.7	566.81	453.26	100.86	1100.2	977.9	101.36	1097.7
0.8	568.74	448.85	100.87	1100.3	969.5	100.93	1097.2
0.9	569.3	445.53	100.71	1100.2	982.9	101.24	1097.3
1	569.91	436.44	100.29	1099.9	975.1	101.15	1097.6

Abbrev.:
1) SBL = solution + bottle + lid
2) BL = bottle + lid
3) -100&R = -100 ml of solution for dilution & -rinse required for pipet wash

Table 3 ~~shows~~ T.G. 7-24-94 TG allows us to determine what volume remains.
Note: The approximate weight of a 500 ml PP Nalgene bottle is: BL ≅ 54.72g

9/8/92 T.G.

After the 0.5, 0.05, and 0.005 total normality strontium solutions were made and weighed, it was decided that the pH of the solutions be weighed. Although the spiked solutions have not yet been made, one can read the b.j procedures on pages 227-228 and see that the pH of these solutions will also be measured. Since our spike comes in a 0.1 M HCl solution, we are expecting the pH to decrease slightly.

The current pH of each solution, w/o spike, was taken and recorded below ~~One might~~ T.G. 7-24-94 TG Analysis of the 0.5 N solutions shows us a fluctuation in the pH. This might possibly be due to the instability TG 7-24-94 of the solutions at high concentrations. The response of the pH electrode was slower than usual.

Solutions' pH Prior to Addition of Sr(90) Spike

0.5 TN E(Sr,i)	pH of 0.5 TN	Temp. (C)	0.05 TN E(Sr,i)	pH of 0.05 TN	Temp. (C)	0.005 TN E(Sr,i)	pH of 0.005 T	Temp. (C)
0.05	5.48	20.4	0.05	----	----	0.05	----	----
0.1	5.67	20.5	0.1	5.54	21.1	0.1	5.64	21.5
0.2	5.54	20.5	0.2	5.55	21.1	0.2	5.65	21.5
0.3	5.55	20.6	0.3	5.52	21.1	0.3	5.67	21.5
0.4	5.51	20.7	0.4	5.58	21.2	0.4	5.64	21.5
0.5	5.56	20.7	0.5	5.58	21.2	0.5	5.65	21.6
0.6	5.44	20.7	0.6	5.55	21.3	0.6	5.64	21.6
0.7	5.48	20.8	0.7	5.57	21.3	0.7	5.67	21.6
0.8	5.49	20.8	0.8	5.57	21.3	0.8	5.64	21.7
0.9	5.51	20.9	0.9	5.59	21.3	0.9	5.68	21.7
1	5.51	21	1	5.63	21.3	1	5.65	21.7

8/14/92 The ^{90}Sr spike was prepared yesterday by Bret Leslie for the addition the the previously made solutions of T.G. TG 7-24-94 0.5, 0.05, and 0.005 NaCl/$SrCl_2$ ~~solutions~~ normality. Today, at 1:30, Dr. Leslie will go over the procedures of adding the spike and the hazards associated with beta ~~emmission~~ TG 7-24-94 TG.

8/14/92 T.G.

0.05 N KCl/$CaCl_2$ Solutions

Purpose: To prepare 0.05 N KCl/$CaCl_2$ solutions using Table 1's weights. We hope our results will show that K/Ca ion exchange is similar to that seen by the Na/Ca isotherm

Reagents: TG 7-24-94
Calcium Chloride Dihydrate (Lot # 4160 KENN)
Potassium Chloride (Lot # 885967)
Several Liters of Nanopure (>17 MΩ·cm resistivity) water

Materials:
AE 240 Mettler Balance (S/N: 101237); Accurate to .1 mg
Beakers
Class A, 1000-ml volumetric flasks
Weighing spatulas
Corning stirrer w/ magnetic stir bar and rod.
TG 7-24-94 ~~Other~~ Wash bottle and eyedropper
30, 60, and 125-ml PP Nalgene bottles

Cont. Procedure:

Preparation of KCl-$CaCl_2$ Solutions

Written by: R. T. Pabalan **Date:** October 13, 1992
Revision No.: 0 **Date Revised:**

1. Prepare 1000 ml of KCl-$CaCl_2$ aqueous mixture with a total normality of 0.05 N and a fixed K/Ca ratio by taring reagent grade KCl and $CaCl_2 \cdot 2H_2O$ in the amounts given in Table 1 onto a clean (acid-washed) 250-ml or 500-ml beaker. Keep the beaker covered with parafilm between additions of reagents to minimize adsorption of water from the atmosphere. Add about 100 ml ultrapure water (>17 megaohm resistivity) to the beaker, add a stir bar and dissolve the salts completely on a stir plate (add more water if necessary). Retrieve the stir bar with a magnetic rod, then carefully rinse the rod and stir bar with ultrapure water, making sure to catch all the washings in the beaker.

Decant the solution into a clean 1000 ml volumetric flask (with the help of a glass rod); rinse the beaker (and glass rod) with ultrapure water several times, carefully transferring all washings into the volumetric flask. Then fill the flask to about 2-3 inches below the mark with ultrapure water; swirl or shake the flask. Let stand for a few seconds, then add water dropwise up to the mark; remix.

Transfer the solution into a clean 1000 ml polypropylene bottle. Label the bottle (e.g., $KCl/CaCl_2$*0.05N*0.1X_{Ca}, plus Date and Initial).

The actual weights are recorded below.

TABLE 1

X_{iCa} (0.05 N) (KCl-$CaCl_2$ soln.)	Wt. $CaCl_2.2H_2O$ needed for 1000 ml	Wt. KCl needed for 1000 ml	Wt. $CaCl_2.2H_2O$ used M.W=147.02	Wt. KCl used M.W=74.56
0.1	0.36754	3.3548	0.36753	3.35489
0.2	0.73508	2.9820	0.73532	2.98188
0.3	1.1026	2.6093	1.10293	2.6094
0.4	1.4702	2.2365	1.4704	2.2366
0.5	1.8377	1.8638	1.8379	1.8639
0.6	2.2052	1.4910	2.2054	1.4909
0.7	2.5728	1.1183	2.57305	1.1828
0.8	2.9403	0.74551	2.94040	0.74558
0.9	3.3079	0.37275	3.30807	0.37282
1	3.6754	0	3.67585	0

8/15/92 T.G.

The following table shows that the given weights required to make the 0.05 N solutions are correct and that by using these weights, one can see that TN ≅ 0.05.

Total Normality Calculations
0.05 N KCl-(CaCl2 DiHyd.) Solutions

Wt.CaCl2 in 1000 m	Wt.KCl in 1000 m	MW.CaCl g/mol	MW.KCl g/mol	Tot.Vol (ml)	M(Ca) mol/L	M(K) mol/L	TN(Na+Sr equiv/L
0.36754	3.3548	147.02	74.56	1000	0.0025	0.044995	**0.049994**
0.73508	2.982	147.02	74.56	1000	0.005	0.039995	**0.049994**
1.1026	2.6093	147.02	74.56	1000	0.0075	0.034996	**0.049995**
1.4702	2.2365	147.02	74.56	1000	0.01	0.029996	**0.049996**
1.8377	1.8638	147.02	74.56	1000	0.0125	0.024997	**0.049997**
2.2052	1.491	147.02	74.56	1000	0.014999	0.019997	**0.049996**
2.5728	1.1183	147.02	74.56	1000	0.0175	0.014999	**0.049998**
2.9403	0.74551	147.02	74.56	1000	0.019999	0.009999	**0.049997**
3.3079	0.37275	147.02	74.56	1000	0.0225	0.004999	**0.049999**
3.6754	0	147.02	74.56	1000	0.024999	0	**0.049999**

8/16/92 T.G.

Using the 0.05 N KCl/$CaCl_2$ solutions prepared on the previous page, the CDV #: KCl/$CaCl_2$ mixtures were made. Using clean acid-washed 30, 60, and 125-ml PP Nalgene bottles, weigh in the amount of zeolite found in Table 1, column 3, after T.G. 8/24-94. The weight of the empty bottle (plus lid) has been recorded in Table 2. Record the weight of the zeolite also.
Note: This CDV is a KF zeolite.

cont. T.6.

That is, on pg 133, Jim Prickril prepared a zeolite whose exchangeable cations were replaced by K^+ cations using a 3M KCl solution. Go to his procedure for more detail.

After each ~~10~~ 24 bottle's weight has been recorded and the ~~10~~ 24 bottles labled as Sample #1-19, and 0.05N KCl/$CaCl_2$ ~~on it~~ then proceed to add the volume of E_i,Ca^{2+} solution found in Table 1. After each volume has been added, cap the bottles and weigh the filled bottles. This should provide the exact solution's weight if sometime later, reverse isotherms would like to be observed.

Table 1

EXPERIMENT KCACL-.05N

CDV*KF

Mixture # KCa05*	$X_{i,Ca}$ to use	Weight zeol to use (gm)	Volume soln to use (ml)	K,i Dilution Factor	Ca,i Dilution Factor	K,f Dilution Factor	Ca,f Dilution Factor
1	0.1	0.3104	25	1/10	10/10	1/10	10/10
2	0.1	0.1662	25	1/10	10/10	1/10	10/10
3	0.1	0.1032	25	1/10	10/10	1/10	10/10
4	0.1	0.0864	50	1/10	10/10	1/10	10/10
5	0.2	0.1518	25	1/10	10/10	1/10	10/10
6	0.2	0.1326	50	1/10	10/10	1/10	10/10
7	0.3	0.1080	25	1/10	10/10	1/10	10/10
8	0.3	0.1187	50	1/10	10/10	1/10	10/10
9	0.4	0.0918	25	1/10	10/10	1/10	10/10
10	0.4	0.0943	50	1/10	10/10	1/10	10/10
11	0.5	0.1402	50	1/10	10/10	1/10	10/10
12	0.6	0.1307	50	10/10	10/10	1/10	10/10
13	0.7	0.1178	50	10/10	10/10	10/10	10/10
14	0.8	0.1131	50	10/10	1/10	10/10	10/10
15	0.9	0.1694	50	10/10	1/10	10/10	10/10
16	0.9	0.1050	50	10/10	1/10	10/10	1/10
17	0.9	0.0640	100	10/10	1/10	10/10	1/10
18	1.0	0.1358	100	--	1/10	10/10	1/10
19	1.0	0.0836	100	--	1/10	10/10	1/10

Should be 1/10 T.G. 10/6/92

cont. T.6.

All of our values from this experiment were recorded in Table 2, which follows.

The dilutions factors found in table 1 will be used later when we determine the amount of solution found in the solutions prepared below.

TABLE 2

Experiment KCACL-0.05N

Solution Preparation

Sample #	Wt.Zeol.	Actual Wt.	E(i,Ca)	Vol.E(Ca)	BL Wt.	SBL+CD	Sol.Wt
1	0.3104	0.3104	0.1	25	8.0374	33.325	24.9772
2	0.1662	0.1663	0.1	25	7.9792	33.1078	24.9623
3	0.1032	0.1032	0.1	25	7.8435	32.9131	24.9664
4	0.0864	0.0864	0.1	50	11.5289	61.5673	49.952
5	0.1518	0.1517	0.2	25	8.0129	33.1333	24.9687
6	0.1326	0.1326	0.2	50	11.5047	61.7104	50.0731
7	0.108	0.108	0.3	25	7.8308	32.9095	24.9707
8	0.1187	0.1187	0.3	50	11.6701	61.8495	50.0607
9	0.0918	0.0919	0.4	25	7.9801	33.0293	24.9573
10	0.0943	0.0943	0.4	50	11.3511	61.6629	50.2175
11	0.1402	0.1403	0.5	50	11.3055	61.4921	50.0463
12	0.1307	0.1307	0.6	50	11.5052	61.5883	49.9524
13	0.1178	0.1178	0.7	50	11.5328	61.6649	50.0143
14	0.1131	0.1132	0.8	50	11.5228	61.5721	49.9361
15	0.1694	0.1696	0.9	50	11.334	61.4639	49.9603
16	0.105	0.1051	0.9	50	11.3759	61.5853	50.1043
17	0.064	0.0641	0.9	100	19.286	119.2493	99.8992
18	0.1358	0.1357	1	100	18.7183	118.7635	99.9095
19	0.0836	0.0835	1	100	19.1291	119.1362	99.9236

BL Wt.: Bottle + Lid Weight

SBL+CDV: Equal Solution + bottle + Lid + CDV amount

8/19/92 T.6.

Each part b. of the procedure found on pg 227 was changed. The change is underlined below.

Procedure for Preparation of NaCl-$^{90}SrCl_2$ Solutions

WRITTEN BY: R.T. Pabalan
REVISION NO.: 1

DATE: September 30, 1992
DATE REVISED: October 14, 1992

1. Prepare 500 ml of NaCl-$SrCl_2$ aqueous mixture with a total normality of 0.5 N and a fixed Na/Sr ratio by taring reagent grade $SrCl_2 \cdot 6H_2O$ in the amount given in Table 1 onto a clean 500 ml beaker. Add about 300 ml deionized water to the beaker, add a stir bar and dissolve the salts completely on a stir plate (add more water if necessary). Retrieve the stir bar with a magnetic rod, then carefully rinse the rod and stir bar with deionized water. Decant the solution into a clean 500 ml volumetric flask (with the help of a glass rod); rinse the beaker (and glass rod) with deionized water several times, carefully transferring all washings into the volumetric flask. Then fill the flask to about 2-3 inches below the mark with deionized water; swirl or shake the flask. Let stand for a few seconds, then add water dropwise up to the mark; remix.

b For each solution prepared above:

On a Mettler AE240 balance, tare a 200 mg aliquot (record the actual weight) of a 0.5 µCi/g ^{90}Sr spike on a plastic boat. On a Mettler PM4600 balance, tare a pre-labeled (e.g., Na/Sr90*0.5N*0.1X_{Sr}, plus Date and Initial) 250-ml polypropylene bottle, and carefully transfer the ^{90}Sr aliquot into the bottle. Rinse the boat with the solution and transfer all washings into the bottle. Transfer a total of 200 g of the NaCl-$SrCl_2$ solution into the bottle (final solution weight=200.20 g). Mix well and measure the pH of the solution. Put a radioactive symbol sticker on the bottle.

Transfer the remaining solutions into clean 500 ml polypropylene bottles. Label the bottle (e.g., Na/Sr*0.5N*0.1X_{Sr}, plus Date and Initial). Measure the pH of each solution.

2. Make up 1000 ml of 0.05N NaCl-$SrCl_2$ solution (except the 0.05X_{Sr} solution) by pipetting 100 ml of the 0.5 N solution and diluting it with deionized water in a 1000 ml volumetric flask. Make sure to initially rinse the pipet with a few ml of the 0.5 N solution.

b For solutions with X_{Sr} equal to 0.1 to 0.9:

On a Mettler AE240 balance, tare a 250 mg aliquot (record the actual weight) of a 0.5 µCi/g ^{90}Sr spike on a plastic boat. On a Mettler PM4600 balance, tare a pre-labeled (e.g., Na/Sr90*0.05N*0.1X_{Sr}, plus Date and Initial) 250-ml polypropylene bottle, and carefully transfer the ^{90}Sr aliquot into the bottle. Rinse the boat with the solution and transfer all washings into the bottle. Transfer a total of 250 g of the NaCl-$SrCl_2$ solution into the bottle (final solution weight=250.25 g). Mix well and measure the pH of the solution. Put a radioactive symbol sticker on the bottle.

For the solution with X_{Sr} equal to 1.0:

Do as in the above paragraph, but use 500 ml of the solution and 500 mg of the

cont. T.6.

^{90}Sr spike (final solution weight = 500.50; in 500-ml polypropylene bottle).

Transfer the remaining 0.05 N solution into 1000 ml polypropylene bottles. Label the bottles (e.g., Na/Sr*0.05N*0.1X_{Sr}, plus Date and Initial). Measure the pH of each solution.

3. Make up 1000 ml of 0.005N NaCl-$SrCl_2$ solution by pipetting 100 ml of the 0.05 N solution and diluting it with deionized water in a 1000-ml volumetric flask. Make sure to initially rinse the pipet with a few ml of the 0.05 N solution.

b For solutions with X_{Sr} equal to 0.1 to 0.9:

On a Mettler AE240 balance, tare a 500 mg aliquot (record the actual weight) of a 0.5 µCi/g ^{90}Sr spike on a plastic boat. On a Mettler PM4600 balance, tare a pre-labeled (e.g., Na/Sr90*0.005N*0.1X_{Sr}, plus Date and Initial) 500-ml polypropylene bottle, and carefully transfer the ^{90}Sr aliquot into the bottle. Rinse the boat with the solution and transfer all washings into the bottle. Transfer a total of 500 g of the NaCl-$SrCl_2$ solution into the bottle (final solution weight=500.50 g). Mix well and measure the pH of the solution. Put a radioactive symbol sticker on the bottle.

For the solution with X_{Sr} equal to 1.0:

Do as in the above paragraph, but use 1000 ml of the solution and 1000 mg of the ^{90}Sr spike (final solution weight = 1001.00; in 1000-ml polypropylene bottle).

Transfer the remaining 0.005 N solution into 500 ml polypropylene bottles. Label the bottle (e.g., Na/Sr*0.005N*0.1X_{Sr}, plus Date and Initial). Measure the pH of each solution.

10/20/92 T.6.

Placed experimental KCaCl-0.05N solutions (mixtures) into a Versa Bath® S, model 236. They will remain in the bath for at least a week while they equilibrate. The temperature of the bath is ~25°C and the RPM's of the shaker bath is 40.

10/20/92 T.6.

Bret Leslie and myself determined the efficiency of the ∗T6. Packard Liquid Scintillation Analyzer by adding 10 µl of the ^{90}Sr spike to 10 ml of each Ei$_{Sr}$ solution. Because the LSA efficiency is dependent of the solution volume, it is imperative that all volumes be nearly the same if

cont. T6. the time counting is the same. When Bobby Pabalan designed this experiment, 7.24.94 T.G. he based ~~most~~ T.G. 7-24-94 his calculations on 25% efficiency for Cherenkov counting. To get precisely 10 ml, we used a 5 ml Oxford Macro-Set pipettor twice. The actual volumes are below.

Volume Accuracy of Oxford Macro-Set

Sample #	BL Wt.	SBL Wt.	Sol.Wt.	Density	Sol.Vol.
1 (0.05)	7.0171	17.2846	10.2675	1.029595	9.972371
2 (0.1)	6.8991	17.171	10.2719	1.030266	9.970146
3 (0.2)	6.8449	17.1056	10.2607	1.031609	9.946303
4 (0.3)	6.8261	17.0855	10.2594	1.032952	9.932113
5 (0.4)	6.9136	17.1806	10.267	1.034297	9.926553
6 (0.5)	6.9533	17.2441	10.2908	1.035641	9.936652
7 (0.6)	6.9228	17.237	10.3142	1.036982	9.946368
8 (0.7)	6.9489	17.2701	10.3212	1.038326	9.940231
9 (0.8)	6.872	17.2305	10.3585	1.03967	9.963254
10 (0.9)	6.8997	17.2349	10.3352	1.041013	9.928023
11 (1.0)	6.932	17.2849	10.3529	1.042356	9.932209
12 (DIW-1)	6.9855	17.0108	10.0253	0.9997	10.02831
13 (DIW-2)	7.0006	17.0415	10.0409	0.9997	10.04391
14 (DIW-3)	6.8663	16.92	10.0537	0.9997	10.05672
				Avg.Vol.:	9.96594

SrCl2.6H2O	% SrCl2	SrCl2 Wt.	Actual NaCl Wt.	Density
1.6669	0.639959	1.066748	13.8806	1.029595
3.3325	0.639959	2.132665	13.1502	1.030266
6.6658	0.639959	4.265842	11.6889	1.031609
9.998	0.639959	6.398315	10.2279	1.032952
13.3315	0.639959	8.53162	8.7667	1.034297
16.6645	0.639959	10.6646	7.3057	1.035641
19.9959	0.639959	12.79657	5.8442	1.036982
23.3291	0.639959	14.92968	4.3833	1.038326
26.6621	0.639959	17.06266	2.9225	1.03967
29.9943	0.639959	19.19514	1.4613	1.041013
33.3273	0.639959	21.32812	0	1.042356

cont. T6.

At ~25% efficiency and knowing the activity of the 20µl spike, it was determined that 5min counting time would be adequate for each vial. The results agreed ~~confirmed~~ T6 7-24-94 to, within Bobby's conjecture of the counting efficiency.

10/20/92 T.G.

Procedure T.6., pg. 236, was performed. Precautions for this experiment must be observed. Ring and chest film, badges were worn, and acryllic shielding capable of stopping beta emmissions T6 7-24-94 were used. Acryllic boxes were used to place the solutions in once made. The weights of the spike and solution total are below.

Preparation of NaCl-90SrCl2 Spiked Reference Solutions

Sample #	Desrcip. of Sol.	Wt. 90Sr Spike	Wt 0.5N w/ 90Sr	Activity @ Surface mRem/hr
1	0.5N,0.05ESr	0.1982	200.22	approx. 0.1
2	0.5N,0.1ESr	0.1992	200.23	approx. 0.1
3	0.5N,0.2ESr	0.1993	200.22	approx. 0.1
4	0.5N,0.3ESr	0.2	200.32	approx. 0.1
5	0.5N,0.4ESr	0.1983	200.23	approx. 0.1
6	0.5N,0.5ESr	0.1992	200.23	approx. 0.1
7	0.5N,0.6ESr	0.2003	202.7	approx. 0.1
8	0.5N,0.7ESr	0.2006	200.21	approx. 0.1
9	0.5N,0.8ESr	0.1993	200.22	approx. 0.1
10	0.5N,0.9ESr	0.1985	200.23	approx. 0.1
11	0.5N,~~0.7~~1.0ESr	0.1994	200.2	approx. 0.1

T6 7-24-94

cont. T6.

Once all the solutions have been made, record the activity of each bottle at the surface, and write it on the bottle, using the durable, radiation hazard stickers and a permant marker. All bottles were placed in acrylic box #.

10/21/92 T.6.

Prepare the CDV-NaSrCl-0.5N mixtures by ~~removing~~ T7-14-94 T.6. adding precalculated and optimized amounts of CDV to T6- predetermined volumes of E(i,Sr solutions. For this part of the experiment, film badges must be worn and the experiment will conducted behind the acrylic shields.

Weigh the 15, 30, and 60-ml acid-washed bottles prior to adding any CDV or solution. Record the bottle and lid weight. Use the weights in column B T6. T6-24-94 and try to accurately add the same amount to the empty bottle. Record the weight. Add the volume in Column D E below using the required equivalent Strontium solution. Record the weight of the entire bottle. When all CDV and solutions have been added to the bottles in the order found in the following chart, place the labeled bottles into a Versa Bath®s, Model 236 for an equilibration period of approximately 10 day. Label the bath as radioactive for passerby's awareness. Clean the work area with DEFENSAFS swips and check for contamination.

cont.

Experiment NaSrCl-0.5N

Solution Preparation w/ 90Sr Spike

Sample #	Wt.Zeol.	Actual Wt.	E(i,Sr)	Vol.E(Sr)	BL Wt.	SBL+CD	Mix.Wt
1	1.529	1.5291	0.05	10	7.2519	18.883	10.102
2	0.1536	0.1534	0.05	10	7.1363	17.3729	10.0832
3	0.3445	0.3445	0.1	10	7.2065	17.6723	10.1213
4	0.7549	0.7549	0.2	10	7.2014	18.0725	10.1162
5	0.1158	0.1159	0.2	25	8.0248	33.3304	25.1897
6	0.3397	0.3398	0.3	10	7.1435	17.613	10.1297
7	0.4992	0.4991	0.4	10	7.0901	17.7025	10.1133
8	0.1424	0.1424	0.4	10	7.0887	17.33	10.0989
9	0.1718	0.1718	0.5	25	7.9973	33.5163	25.3472
10	0.08567	0.0857	0.6	10	7.11	17.3975	10.2018
11	0.1583	0.1583	0.7	10	7.1075	17.4569	10.1911
12	0.1858	0.1858	0.8	10	7.2191	17.6017	10.1968
13	0.3184	0.3184	0.9	10	7.0555	17.5529	10.179
14	0.1338	0.1337	0.9	10	7.1238	17.4193	10.1618
15	0.3232	0.3232	1	10	6.9746	17.49	10.1922
16	0.221	0.221	1	10	7.0795	17.4819	10.1814
17	0.1477	0.1477	1	10	6.9014	17.2307	10.1816
18	0.1963	0.1963	1	25	7.9149	33.6692	25.558
19	0.116	0.116	1	50	11.4198	62.4776	50.9418
column	A	B	C	D	E	F	G	H

T6-24-94

Abbrev.:

BL Wt.: Bottle + Lid weight

SBL+CDV: E(i,Sr) Solution + Bottle + Lid + CDV amount

T6-24-94

* The volumes in column ~~D~~ E were added using the Oxford Macro-Set pipettor with disposable tips. Recall from page 238 that this is a 5ml adjustable pipettor and consequently, it ~~with~~ T6. was used several times for the solutions. 3 different tips were used for the 60 Esr solution since large volumes were used and I wanted to avoid wetting on the pipet surfaces.

10/5/92 T.G.

On the 28th of October, the 0.05 N KCl-$CaCl_2$ solutions were removed from the Versa Water bath. These mixtures were diluted according to the dilution factors found on page 234. The first attempts at measuring the K^+ concentrations were on 9/23/92; however, I became ill and was unable to log my data (it was stored in a computer). Today, I simply measured the same mixture bottles using new standards and freshly prepared electrodes. Results from both days are below.

K+ Measurements

Sample #	1st Try.K	2nd Try.K	3rd Try.K	4th. K+	'5th. K+	6th. K+	Avg.K+
1	174			170		175	1730
2	174			166		173	1710
3	169			162		170	1670
4	167			159		166	1640
5	146			142		149	1456.667
6	149			143		148	1466.667
7	128			124	128	128	1270
8	127			120	128	130	1262.5
9	*109	**106	113		112	111	672
10		115			111		1130
11		94.5			92.4		934.5
12		75.8			75.4		756
13		610			626		618
14		416	425		430		423.6667
15		252	273		265		263.3333
16		236	249		246		243.6667
17		207	218		221		215.3333
18		57.2	58.7		60.8		58.9
19		38.4	41.7		41.8		40.63333
Date:	9-29-92	9-29-92	9-29-92	10-5-92	10-5-92	10-5-92	
Slope(mV	57.5	57.2	56.9	56.4	56.8	57.5	

*(Drift): 100 ppm Std. read 90.7
**(Drift): 100 ppm Std. read 89.9

cont. For the mixtures (T.G. 7-24-94) solutions used in K^+ analysis, a 0.2 ml (T.G.) aliquot of 5M KCl, which is simply (T.G. 7-24-94) for ionic strength adjustor. The solutions were measured at a fairly (T.G. 7-24-94) constant room temperature. The first 12 K^+ solutions were diluted by 1/10 using Class A 10-ml volumetrics. All solutions were placed in 15 (T.G.) ml PP Nalgene bottles.

10/6/92 T.G.
Set up Calcium Ion Selective Electrodes for solution measurements. Began measuring these solutions, but (T.G. 7-24-94) on the same day. Note that these solutions were prepared on 9/28/92 when the K^+ solutions were prepared; however, they were set aside until a later date for which they could be measured.
Each solution had 0.2 ml of (4th T.G. 10/6/92) KCl solution added to the 10-ml PP bottles in order to adjust the ionic strength of the (T.G. 7-24-94) solution. The dilution factor of each solution can be found on pg 234.
Below are the results.

Sample#	1st. Ca+2
1	79.6
2	82.8
3	82.8
4	86
5	171
6	173
7	258
8	264
9	353
10	362
11	456
12	550
13	641
14	750
15	825
16	82.7
17	83.1
18	89.9
19	91
Date:	10-6-92
Slope(mV	28.8

10/9/92 T.G.

The references were measured to determine the Ca^{2+} concentration 'actually' added. I use the term 'actually' very loosely, since on occassions, the electrode response is slow and the drift relatively high. I expect the Ca^{2+} concentration to be lower than expected, which is what is seen below in the results. An obvious explanation for this ~~is that when~~ T.G 1-14-94 arises from the fact that when $CaCl_2 \cdot 2H_2O$ (Calcium Dihydrate) is open to air, especially high humidity air, water absorbs onto the calcium crystal thereby liquifying it and ~~gaining~~ T.G 1-24-94 gaining weight. The concentrations were recorded and are listed below.

Ca+2 Reference Results

E(i,Ca+2)	1st Ca+2	2nd Ca+2	3rd Ca+2	Dil.Fact.	Avg. Ca+
0.1		88.7		10/10	88.7
0.2	177	180		10/10	178.5
0.3	266	270		10/10	268
0.4	362	365		10/10	363.5
0.5	460	459		10/10	459.5
0.6	546	549	556	10/10	550.3333
0.7	65.9	67	67.7	1/10	668.6667
0.8	73.3	77.7	77.7	1/10	762.3333
0.9	83.1	87.6	86.9	1/10	858.6667
1	93.1	97.1	96.4	1/10	955.3333
Date:	10-9-92	10-9-92	10-9-92		
Slope(mV	28.6	28.9	28.8		

11/15/92 T.G.

The references were measured to determine the K^+ concentration using a ~~Elmer~~ T.G 7-24-94 Perkin & Elmer Model 3100 Atomic Absorption Spectrometer. For these measurements, a red filter must be used to eliminate the lower wavelength interferences. Two wavelengths were used. The first wavelength worked in a linear ppm range of >0-2. These results are listed below, but are unattractive. KCl is not the type of salt to pull a lot of water from the air, and the salt has no water to lose to the air. However, AA results using wavelength consistently and proportionally show a higher than expected value. Therefore, Jim Prikryl and I tryed a different wavelength which worked in the linear range of 4-20 ppm. These results ~~were~~ are T.G 7-24-94 also listed below.

2 ppm ≈ 0.258 Abs. Absorbance

E(i,Ca+2)	1st,K+	2nd,Ab	2nd,K+	1st,Actual	2nd,Actual	Linear Range of 4-20 Results using 3rd,K+	4th,K+	3rd,Actu	4th,Actu	Avg 3 & 4 K+
0.1	1.84	0.238	1.836	1840	1835.71	17.78	17.6	1778	1760	1769
0.2	1.612	0.223	1.72	1612	1720.02	15.64	15.65	1564	1565	1564.5
0.3	1.46	0.192	1.481	1460	1480.91	13.74	13.71	1374	1371	1372.5
0.4	1.343	0.158	1.219	1343	1218.67	11.69	11.63	1169	1163	1166
0.5		0.129	0.995	0	994.987	9.74	9.78	974	978	976
0.6		0.115	0.887	0	887.003	7.61	7.72	761	772	766.5
0.7	0.599	0.08	0.617	599	617.046	5.72	5.68	572	568	570
0.8	0.501	0.056	0.432	501	431.932	3.73	3.78	373	378	375.5
0.9	0.37	0.031	0.239	370	239.105	19.46	19.49	194.6	194.9	194.75
1	0	0	0	0	0	0	0	0	0	0

Cont.

Because the first attempt using the Ag resulted in high values, I decided to use an addition method. The output from this method is absorbance and it needs to be converted into ppm's. Therefore, write down the absorbance values when calibrating with the standard. The 2 ppm K^+ std was prepared from a 1000 ppm Potassium Reference Solution, Lot #910846-18. The absorbance of the 2 ppm Std was found to be 0.258. Knowing the absorbance of any other solution, the ppm conc. can be determined.

e.g. A 0.238 abs is:

$$0.238 * \frac{2 \text{ ppm}}{0.258 \text{ abs}} = 1.836$$ (See previous page).

I believe that the higher linear range worked better, even though sensitivity is lost in this range. This increase in accuracy is due to a better dilution factor. In the 0-2 ppm range, the dilution must be 1/1000 or 0.1 ml in 100 ml (NOT VERY ACCURATE). In the 4-20 ppm range, the dilution factor is 1/100 and glass pipets were used.

In conclusion, the results are better representatives of the true concentrations when in the 4-20 ppm linear range.

10/6/92 For the above K^+ analyses, 0.1% Lanthanum Chloride was used to stop interferences, not cesium chloride. "

10/11/92 T.G.

After determining that the atomic absorption spectrometer provided decent results for the K^+ reference measurements, I decided to try our Hollow Cathode Lamp for Ca^{2+}-Mg^{2+}. The lamps can be used for two elements. The 5ppm Ca^{2+} std was prepared from a 1000 ppm Calcium Standard (Rica Chem. Co.), Lot # C076. Using the same reference STD, prepare a 4 ppm calibration standard as a check. Use a 0.1% KCl solution to prepare the dilutions and stop ionization interferences.

Jim Pitkyl and I couldn't seem to stabilize the absorbance and maximize the energy. We therefore decided to lower the energy and gain in an attempt to stabilize the absorbance. Neither proved successful. Measuring the solutions anyways showed that the absorbance wasn't stable. The range in ppm's demonstrates that. The results follow.

AA Concentrations of 0.05 N KCl-CaCl2 Solutions

E(i,Ca+2)	1st, Ca+2	2nd, Ca+	Dil.factor	1st,Actual	2nd,Actua
0.1	0.066	0.022	1000	66	22
0.2	0.184	0.133	1000	184	133
0.3	0.307	0.251	1000	307	251
0.4	0.417	0.361	1000	417	361
0.5	0.531	0.468	1000	531	468
0.6	0.624	0.571	1000	624	571
0.7	0.703	0.662	1000	703	662
0.8	0.8	0.759	1000	800	759
0.9	0.865	0.836	1000	865	836
1	1.046	1.05	1000	1046	1050

TG 7.24.94

10/12/92 T.G.

I again got out the Calcium ion selective electrode to check the previous results recorded on 10/6/92. New solutions, standards, were prepared. For convenience, the data resulting from the first analyses on 10/6/92 are also listed below.

Ca+2 Concentrations

Sample #	1st. Ca+2	2nd.Ca+2	3rd.Ca+2	4th.Ca+2	Dil.Fact.	Avg.Ca+2
1	79.6	78.9	81.8		10/10	80.1
2	82.8	80.8	84.1		10/10	82.56667
3	82.8	82.2	85.4		10/10	83.46667
4	86	81.9	86.2		10/10	84.7
5	171	163	172		10/10	168.6667
6	173	166	173		10/10	170.6667
7	258	250	259		10/10	255.6667
8	264	250	262		10/10	258.6667
9	353	337	352		10/10	347.3333
10	362	339	357		10/10	352.6667
11	456	426	452		10/10	444.6667
12	550	524	549		10/10	541
13	641	623	633	637	10/10	633.5
14	750	732		738	10/10	740
15	825	826		818	10/10	823
16	82.7	81.5		85.1	1/10	83.1
17	83.1	82.2		86.1	1/10	83.8
18	89.9	89.8		93.2	1/10	90.96667
19	91	89.8		94.3	1/10	91.7
Date:	10-6-92	10-12-92	10-12-92	10-12-92		
Slope(mV	28.8	28.7	28.8	28.6		

TG 7-24-94

10/13/92 T.G.

After analyzing the 'actual' Ca^{2+} concentrations found on page 247, I decided to again measure the reference solutions. This time however, the dilution factor varied from 1/100 to 1/250 instead of ~~1~~ 1/1000 (0.1ml in 100ml). Each solution was measured twice -- once as ppm and the other as absorbance using known addition. In both cases, the results from the solutions showed high values which ~~was~~ is unreasonable. Another 5ppm std was made to use as a reslope check. Both solutions were measured and within 2% of each others concentration. A 4ppm std was prepared as a check for the slope and after a 5ppm calibration, the 4 ppm std read 4.123 ppm. A serial dilution from the 5 ppm std was ~~made~~ performed TG 7-24-94 in order to make a 1ppm solution and the result ~~was~~ was TG 7-24-94 1.201 ppm. Analysis of the stds and solutions shows that the slope is consistently off.

E(i,Ca+2)	1st,Ca+2	Actual	Abs.	Abs.Act.(pp	Dil.Fact.	Abs(ppm)
0.1	1.208	120.8	0.077	1.170213	1/100	117.0213
0.2	2.211	221.1	0.145	2.203647	1/100	220.3647
0.3	3.219	321.9	0.212	3.221884	1/100	322.1884
0.4	4.19	419	0.277	4.209726	1/100	420.9726
0.5	5.087	508.7	0.337	5.121581	1/100	512.1581
0.6	2.645	661.25	0.174	2.644377	1/250	661.0942
0.7	3.02	755	0.199	3.024316	1/250	756.079
0.8	3.433	858.25	0.227	3.449848	1/250	862.462
0.9	3.794	948.5	0.25	3.799392	1/250	949.848
1	4.21	1052.5	0.278	4.224924	1/250	1056.231

5 ppm = 0.329 abs.
4 ppm check = 4.123 ppm on AA
Dilution from a 5 ppm standard to 1 ppm read 1.201 ppm on AA

11/16/92 T.G.

Corrected all the dates from pg's 248-249.
2
T.G.

11/17/82 KDP

The following zeolite samples were sent to Texas Tech Univ. for ICP analysis.

Sample #	Description
NAKCL5-1	Expt Na/K/Cl 0.5N; E_{K+} = .01
NAKCL5-3	Expt Na/K/Cl 0.5N; E_{K+} = .03
NAKCL5-11	Expt Na/K/Cl 0.5N; E_{K+} = 0.2
NAKCL5-17	Expt Na/K/Cl 0.5N; E_{K+} = 0.6
NAKCL05-1	Expt Na/K/Cl 0.05N; E_{K+} = .01
NAKCL05-5	Expt Na/K/Cl 0.05N; E_{K+} = .075
NAKCL05-9	Expt Na/K/Cl 0.05N; E_{K+} = .15
NAKCL05-19	Expt Na/K/Cl 0.05N; E_{K+} = .75
NACACL5-1	Expt Na/Ca/Cl 0.5N; E_{Ca++} = .02
NACACL5-5	Expt Na/Ca/Cl 0.5N; E_{Ca++} = .1
NACACL5-7	Expt Na/Ca/Cl 0.5N E_{Ca++} = .15
NACACL5-11	Expt Na/Ca/Cl 0.5N E_{Ca++} = .4
NACACL5-13	Expt Na/Ca/Cl 0.5N E_{Ca++} = .6
NACACL05-1	Expt Na/Ca/Cl .05N E_{Ca++} = .015
NACACL05-5	Expt Na/Ca/Cl .05N E_{Ca++} = .1
NACACL05-9	Expt Na/Ca/Cl .05N E_{Ca++} = .3
NACACL05-11	Expt Na/Ca/Cl .05N E_{Ca++} = .5
NACACL05-19	Expt Na/Ca/Cl .05N E_{Ca++} = .9
NAKNO3-1	Expt Na/K/NO_3 .05N E_{Cat+} =
NAKNO3-7	Expt Na/K/NO_3 .05N E_{Cat+} =
NAKNO3-10	Expt Na/K/NO_3 .05N E_{Cat+} =
NAKNO3-14	Expt Na/K/NO_3 .05N E_{Cat+} =
NAKNO3-18	Expt Na/K/NO_3 .05N E_{Cat+} =
NACANO3-1	Expt Na/Ca/NO_3 .05N E_{Cat+} =
NACANO3-7	Expt Na/Ca/NO_3 .05N E_{Cat+} =
NACANO3-10	Expt Na/Ca/NO_3 .05N E_{Cat+} =
NACANO3-14	Expt Na/Ca/NO_3 .05N E_{Cat+} =
NACANO3-18	Expt Na/Ca/NO_3 .05N E_{Cat+} =

KDP 11/17/82

Over

Sample #	Description
CRVKF-1	same as Sample F (CRC-04-155)
CRVKF-2	" " K "
CRVNAF-1	" " C "
CRVNAF-2	" " N "
CRVKF-1	" " E "
CRVKF-2	" " M "
CRVCAF-1	" " O "
CRVCAF-2	" " L "

The following clays were sent for analysis:

Sample #	Description
SCA-3-1	Montmorillonite, Otay, Source Clay Min. Rep.
SCA-3-2	" " " " " "
API24-1	" , Otay, Ward's
API24-2	" , " , "
API24NA-1	" , " , " , Na-Form
API24NA-2	" , " , " , Na-Form
SAZ-1-1	" , Choate, Source Clay Min. Rep.
SAZ-1-2	" , " , " " " "
SAZ-1-3	" , " , " " " "
STX-1-1	" , Gonzales Co, TX, " " " "
STX-1-2	" , " , " , " " " "

The following are NBS standards sent for DCP analysis:

Sample #	Description
NAKCL5-25	NBS 70A
NACACL5-25	NBS 99A
NAKNO3-25	NBS 70A
NACANO3-25	NBS 99A
SST-1	NBS 70A
CRVST-1	NBS 99A

In addition, 2 clearly labeled NBS 99A & NBS 70A were submitted and requested to be run every 10 to 15 unknown samples.

11/25/92 T.6.

At approximately 9:00 a.m., the 0.5 N $Na^{85}Sr$ solutions that had been equilibrating had 1 ml. aliquots ~~di~~ removed from the mixtures. Each 1 ml. was diluted to 10 ml, by ~~weight~~ volume. ~~However~~, Technically, volumetric flasks ~~are~~ were used for proper dilution. Instead, the 1 ml aliquot was added to 9 ml of H_2O and the weights of ~~all~~ the empty LSA vials were recorded and ~~after~~ each subsequent additions of known volumes were also recorded below.

0.5 N Strontium Analyses Using LSA

Sample #	LSA Vial Empty	Vial + 9 ml H2O	Vial+H2O +1ml Sol'	Weight 9 ml H2O	Weight 1 ml Sol'n
1	6.824	15.8498	16.8607	9.0258	1.0109
2	6.9506	15.9425	16.9618	8.9919	1.0193
3	6.9646	15.9431	16.9588	8.9785	1.0157
4	6.8296	15.8063	16.8186	8.9767	1.0123
5	6.9869	15.9872	17.005	9.0003	1.0178
6	6.8478	15.8489	16.8656	9.0011	1.0167
7	7.0292	16.0293	17.0477	9.0001	1.0184
8	6.8666	15.8644	16.8829	8.9978	1.0185
9	6.9887	15.9921	17.0136	9.0034	1.0215
10	6.9634	15.9682	16.989	9.0048	1.0208
11	6.9228	15.9258	16.9502	9.003	1.0244
12	6.8764	15.8883	16.9138	9.0119	1.0255
13	7.0421	16.0398	17.0702	8.9977	1.0304
14	6.8622	15.8645	16.8866	9.0023	1.0221
15	6.9835	15.9886	17.0135	9.0051	1.0249
16	6.9006	15.9019	16.928	9.0013	1.0261
17	6.8547	15.858	16.8841	9.0033	1.0261
18	6.8464	15.8568	16.883	9.0104	1.0262
19	6.9999	16.0068	17.0368	9.0069	1.03

cont.

0.5 90Sr Reference Analyses Using LSA

E(i,Sr)	Vial Wt. Empty	Vial + 10ml Sol'	Weight 10ml Sol'
0.05	6.929039	17.0829	10.15386
0.1	6.9219	17.133	10.2111
0.2	6.9375	17.153	10.2155
0.3	7.0025	17.2409	10.2384
0.4	7.0106	17.2688	10.2582
0.5	6.9408	17.2221	10.2813
0.6	6.9458	17.2706	10.3248
0.7	6.9839	17.2917	10.3078
0.8	6.8937	17.2217	10.328
0.9	6.8375	17.1742	10.3367
1	6.875	17.1647	10.2897

12/1/92 T.6.

After placing the vials previously discussed into the Liquid Scintillation Analyzer, the following results were obtained. Each vial remained in the photomultiplier tube for a 10minute counting time. Questions arising from preparation of the buffer in the LSA and result ~~explanations~~ analyses should be directed toward Bret Leslie. All results can be found on the next page.

cont. T.G.

Results from LSA for 0.5 N 90Sr Solutions

Sample #	Time(min)	CPMA	CPMB	CPMC	SIS
1	10.00	102.4	9.4	111.8	44.259
2	10.00	639.6	11.5	651.1	33.728
3	10.00	544.3	12.7	557	34.504
4	10.00	445.3	11.7	457	34.458
5	10.00	824.4	14.1	838.5	33.334
6	10.00	678.9	13.5	692.4	33.616
7	10.00	639.2	14.6	653.8	34.507
8	10.00	769.3	14.5	783.8	33.323
9	10.00	823.1	16.6	839.7	34.231
10	10.00	825.2	14.6	839.8	33.322
11	10.00	794.1	14.3	808.4	33.084
12	10.00	799.6	14	813.6	33.646
13	10.00	769.5	13	782.5	33.483
14	10.00	824.7	15.4	840.1	33.377
15	10.00	740	13.1	753.1	33.905
16	10.00	779.2	14.8	794	33.621
17	10.00	807.7	13.7	821.4	33.095
18	10.00	823.2	13.1	836.3	33.478
19	10.00	857.6	14.4	872	33.499

Results from LSA for 0.5 N 90Sr Reference Solutions

E(i,Sr+2)	Time(min)	CPMA	CPMB	CPMC	SIS
0.05	10.00	8496	51.4	8547.4	31.529
0.1	10.00	8411.7	63.1	8474.8	31.564
0.2	10.00	8467.7	58.2	8525.9	31.589
0.3	10.00	8500.3	58.6	8558.9	31.588
0.4	10.00	8479	56.5	8535.5	31.553
0.5	10.00	8556.2	56.5	8612.7	31.499
0.6	10.00	8513.3	59.2	8572.5	31.497
0.7	10.00	8637	61.6	8698.6	31.481
0.8	10.00	8603.9	53	8656.9	31.104
0.9	10.00	8600.9	56.7	8657.6	31.389
1	10.00	8516.2	59.1	8575.3	31.294

12/3/92 T.G.

In preparation for measuring the Na^+ in the 0.5N $SrCl_2$-NaCl solutions with the ion selective meter, dilutions were made corresponding to the factors recommended by Bobby Pabalan in September, 1992. These factors can be found below. Note that the Sr,f dilution factors were ~~prepared~~ performed on 11/25/92 along with the Sr,i solutions. TG 7-24-94 TG 7-24-94

EXPERIMENT NASRCL-.5N

Mixture # NaSr5*	$X_{i,Sr}$ to use	Weight zeol. to use (g)	Vol. soln to use (ml)	Na,i Dilution Factor	Sr,i Dilution Factor	Na,f Dilution Factor	Sr,f Dilution Factor
1	0.05	1.5290	10	1/100	10/10	1/100	1/10
2	0.05	0.1536	10	1/100	10/10	1/100	1/10
3	0.1	0.3445	10	1/100	10/10	1/100	1/10
4	0.2	0.7549	10	1/100	10/10	1/100	1/10
5	0.2	0.1158	25	1/100	10/10	1/100	1/10
6	0.3	0.3397	10	1/100	10/10	1/100	1/10
7	0.4	0.4992	10	1/10	10/10	1/10	1/10
8	0.4	0.1424	10	1/10	10/10	1/10	1/10
9	0.5	0.1718	25	1/10	10/10	1/10	1/10
10	0.6	0.08567	10	1/10	10/10	1/10	1/10
11	0.7	0.1583	10	1/10	10/10	1/10	1/10
12	0.8	0.1858	10	1/10	10/10	1/10	1/10
13	0.9	0.3184	10	1/10	10/10	1/10	1/10
14	0.9	0.1338	10	1/10	10/10	1/10	1/10
15	1.0	0.3232	10	1/10	10/10	1/10	1/10
16	1.0	0.2210	10	--	10/10	1/10	1/10
17	1.0	0.1478	10	--	10/10	1/10	1/10
18	1.0	0.1963	25	--	10/10	1/10	1/10
19	1.0	0.1160	50	--	10/10	1/10	1/10

September 30, 1992 TGP

Cont. T.G.

According to the amounts weighed on pg 228 we can deduce an expected concentrations for both Strontium and Sodium. The actual concentrations will most likely be measured tomorrow to determine the actual concentrations in the reference solutions and CDV mixtures.

However, only sodium will be measured since we don't have a strontium hollow cathode lamp to measure the non-radioactive reference solutions. Below are the expected ppm/L concentrations for both Sr^{2+} and Na^{+}.

Expected Results from 0.5 N SrCl2-NaCl References

E(i,Sr+2)	Wt. Sr+2 (grams)	Wt. Na+ (grams)	Amount Sr(ppm/L)	Amount Na(ppm/L	Dil.Fact. Sr+2	Dil.Fact. Na+	Expect Sr+2	Expect Na+
0.05	1.6669	13.8806	1095.595	10921.01	10/10	1/100	1095.6	109.21
0.1	3.3325	13.1502	2190.336	10346.34	10/10	1/100	2190.34	103.463
0.2	6.6658	11.6889	4381.197	9196.616	10/10	1/100	4381.2	91.9662
0.3	9.998	10.2279	6571.336	8047.128	10/10	1/100	6571.34	80.4713
0.4	13.3315	8.7667	8762.329	6897.482	10/10	1/10	8762.33	689.748
0.5	16.6645	7.3057	10952.99	5747.993	10/10	1/10	10953	574.799
0.6	19.9959	5.8442	13142.61	4598.111	10/10	1/10	13142.6	459.811
0.7	23.3291	4.3833	15333.4	3448.702	10/10	1/10	15333.4	344.87
0.8	26.6621	2.9225	17524.07	2299.37	10/10	1/10	17524.1	229.937
0.9	29.9943	1.4613	19714.2	1149.725	10/10	1/10	19714.2	114.972
1	33.3273	0	21904.87	0	10/10	1/10	21904.9	0

Note that the dilutions factors vary for sodium so that the concentration remains below our 1000 ppm Na^{+} Standard.

12-11-92 T.G.

12/3/92 T.G.

The References didnt have spike. Note: two sets of Ref. solutions: One w/spike, one w/o.

T.G. 12-11-92

In making the Na^{+} Solution Reference dilutions for analyses, the two dilutions were performed using glass volumetrics. The elapsed time for glass-^{90}Sr contact was less than 5 min each in order to prevent sorption onto the glass surface, thereby contaminating are acid-bath and other glassware.

The amount of ^{90}Sr dumped down the drain is believed to be less than:

$$508.54 \times 10^{-9} \frac{Ci}{g} * \frac{.2\ ml}{200\ ml} * .5g * \frac{1\ ml}{100\ ml} = 2.54 \times 10^{-12}\ Ci$$

or

2.54 pCi

12/10/92 T.G.

Reverse isotherms may be required in the near future and consequently, the solution volume remaining must be known. For this reason, the amount of solution used for each dilution was weighed after ~~it~~ TG 7-24-94 being removed from each CDV-solution mixture. Recall from the dilution factors on page 257 that each mixture must lose at least two milliliters of solution in order to determine the concentrations of both strontium and sodium. For convenience in calculating the remaining volume weight, the table on page 241 was placed on the following page.

cont.

Experiment NaSrCl-0.5N

Solution Preparation w/ 90Sr Spike

T.G. 12-11-92 Solution wt. of 0.5N

Sample #	Wt.Zeol.	Actual Wt.	E(i,Sr)	Vol.E(Sr)	BL Wt.	SBL+CD	Mix.Wt
1	1.529	1.5291	0.05	10	7.2519	18.883	10.102
2	0.1536	0.1534	0.05	10	7.1363	17.3729	10.0832
3	0.3445	0.3445	0.1	10	7.2065	17.6723	10.1213
4	0.7549	0.7549	0.2	10	7.2014	18.0725	10.1162
5	0.1158	0.1159	0.2	25	8.0248	33.3304	25.1897
6	0.3397	0.3398	0.3	10	7.1435	17.613	10.1297
7	0.4992	0.4991	0.4	10	7.0901	17.7025	10.1133
8	0.1424	0.1424	0.4	10	7.0887	17.33	10.0989
9	0.1718	0.1718	0.5	25	7.9973	33.5163	25.3472
10	0.08567	0.0857	0.6	10	7.11	17.3975	10.2018
11	0.1583	0.1583	0.7	10	7.1075	17.4569	10.1911
12	0.1858	0.1858	0.8	10	7.2191	17.6017	10.1968
13	0.3184	0.3184	0.9	10	7.0555	17.5529	10.179
14	0.1338	0.1337	0.9	10	7.1238	17.4193	10.1618
15	0.3232	0.3232	1	10	6.9746	17.49	10.1922
16	0.221	0.221	1	10	7.0795	17.4819	10.1814
17	0.1477	0.1477	1	10	6.9014	17.2307	10.1816
18	0.1963	0.1963	1	25	7.9149	33.6692	25.558
19	0.116	0.116	1	50	11.4198	62.4776	50.9418

Abbrev.:

BL Wt.: Bottle + Lid weight

SBL+CDV: E(i,Sr) Solution + Bottle + Lid + CDV amount

For clarification purposes, I refer to mixture as being the reference solution plus the CDV. The solution is what is removed after ion exchange has occured. The following page shows the weight of solution removed for preparing each $^{90}Sr^{+2}$ and Na^{+} solutions for measurement. One might note that column 6 on pg 261 should be equal to that found on page 254, col 6. Column 8 on page 261 should equal column 10 on page 262. None of the column weights are equal; however, they are T.G. close.

cont, t.o.

Amount of 0.5 N 90SrCl2-NaCl Solution Used and Remaining

Sample #	BL Wt.	SBL+CD	Mix.Wt	-1ml,90Sr Analyses	Wt.Used 90Sr+2	-2nd ml,N Analyses	Wt.Used Na+
1	7.2519	18.883	11.6311	17.8665	1.0165	16.8497	1.0168
2	7.1363	17.3729	10.2366	16.3437	1.0292	15.3236	1.0201
3	7.2065	17.6723	10.4658	16.6464	1.0259	15.6229	1.0235
4	7.2014	18.0725	10.8711	17.0463	1.0262	16.0262	1.0201
5	8.0248	33.3304	25.3056	32.3207	1.0097	31.2817	1.039
6	7.1435	17.613	10.4695	16.5818	1.0312	15.5461	1.0357
7	7.0901	17.7025	10.6124	16.6717	1.0308	15.6546	1.0171
8	7.0887	17.33	10.2413	16.3207	1.0093	15.2771	1.0436
9	7.9973	33.5163	25.519	32.4856	1.0307	31.4586	1.027
10	7.11	17.3975	10.2875	16.3669	1.0306	15.3403	1.0266
11	7.1075	17.4569	10.3494	16.3603	1.0966	15.3024	1.0579
12	7.2191	17.6017	10.3826	16.5683	1.0334	15.5344	1.0339
13	7.0555	17.5529	10.4974	16.5145	1.0384	15.4811	1.0334
14	7.1238	17.4193	10.2955	16.3824	1.0369	15.3463	1.0361
15	6.9746	17.49	10.5154	16.4124	1.0776	15.3659	1.0465
16	7.0795	17.4819	10.4024	16.4486	1.0333	15.4141	1.0345
17	6.9014	17.2307	10.3293	16.1677	1.063	15.1221	1.0456
18	7.9149	33.6692	25.7543	32.6299	1.0393	31.5971	1.0328
19	11.4198	62.4776	51.0578	61.4316	1.046	60.4025	1.0291

Abbrev.:

BL Wt.: Bottle + Lid weight

SBL+CDV: E(i,Sr) Solution + Bottle + Lid + CDV amount

-3rd ml, Na^{+} Analyses	Wt. Used Na^{+}
15.8298	1.0199
14.3083	1.0153
14.6053	1.0176
15.0099	1.0163
30.2639	1.0178
14.5239	1.0222

T.G. 12-24-92 A 3rd ml was removed from the solutions ~~of~~ T.G. to 7-24-94 1-6 for analyses using a dilution factor of 1/10 instead of 1/100. The values from the ISE meter for the 1/100 dilutions were unrealistic. Therefore, the solutions made on 12/3/92, pg 259, weren't used.

Cont. T.G.

0.5 N Sodium Analyses Preparation

Adjusted TG 7-24-97

Sample #	Vial Wt. Empty	Vial Wt.+ 9 ml H2	Weight 9 ml H2O	New Vial 9 ml H2O	New Wt. 9 ml H2	New Wt. ISA add	Actual ISA adde	New Wt. 1 ml 90S	Actual wt. 90Sr
1	6.9562	15.8726	8.9164	15.9642	9.008	16.9745	1.0103	17.9896	1.0151
2	6.8485	15.9733	9.1248	15.8479	8.9994	16.8606	1.0127	17.872	1.0114
3	6.8841	15.8686	8.9845	15.8934	9.0093	16.9084	1.015	17.9238	1.0154
4	6.8732	15.8744	9.0012	15.8772	9.004	16.8919	1.0147	17.9055	1.0136
5	6.9669	15.8343	8.8674	15.9715	9.0046	16.9832	1.0117	17.9971	1.0139
6	6.8203	15.9497	9.1294	15.8209	9.0006	16.839	1.0181	17.8523	1.0133
7	6.8527	15.9079	9.0552	15.858	9.0053	16.8629	1.0049	17.8781	1.0152
8	6.9273	15.9419	9.0146	15.9311	9.0038	16.9379	1.0068	17.9623	1.0244
9	6.861	15.8688	9.0078	15.867	9.006	16.8753	1.0083	17.9004	1.0251
10	6.9975	16.007	9.0095	16.0061	9.0086	17.0157	1.0096	18.04	1.0243
11	6.8842	15.9057	9.0215	15.885	9.0008	16.8957	1.0107	17.9219	1.0262
12	6.9002	15.9456	9.0454	15.9036	9.0034	16.9157	1.0121	17.946	1.0303
13	6.8329	15.8539	9.021	15.8349	9.002	16.8459	1.011	17.8768	1.0309
14	6.9217	15.9496	9.0279	15.929	9.0073	16.9396	1.0106	17.9703	1.0307
15	6.965	15.9865	9.0215	15.9667	9.0017	16.9785	1.0118	18.011	1.0325
16	6.8334	15.8825	9.0491	15.8422	9.0088	16.8543	1.0121	17.8863	1.032
17	6.8866	15.9353	9.0487	15.895	9.0084	16.9054	1.0104	17.9381	1.0327
18	6.8958	15.9312	9.0354	15.9008	9.005	16.9114	1.0106	17.943	1.0316
19	6.9028	15.9543	9.0515	15.9002	8.9974	16.9108	1.0106	17.9334	1.0226

The sodium analyses solutions were made using nearly the same method as the strontium analyses solutions. However, each vial was adjusted using micropipettors by Eppendorf. TG 7-24-97 In this T.G. way, Adjusting each vial to 9 ml ensures that all solutions will have nearly the same dilution. The results from the references and the solutions are found on the next page for sodium analyses.

Cont. T.G.

Actual Measurements of 0.5 N SrCl2-NaCl References & Solutions

References

E(i,Sr+2)	Dil.Factor	Na+		Dil. Factor	Na+	Actual N
0.05	1/100	103		1/10	1110	11100
0.1	1/100	95.1		1/10	1040	10400
0.2	1/100	82.9		1/10	918	9180
0.3	1/100	94.5		1/10	802	8020
0.4	1/10	682		1/10	682	6820
0.5	1/10	573		1/10	573	5730
0.6	1/10	457		1/10	457	4570
0.7	1/10	343		1/10	343	3430
0.8	1/10	229		1/10	229	2290
0.9	1/10	109		1/10	108	1080
1	1/10	0		1/10	0	0
Slope:		60.2			59.8	
Date		10/8/92			10/8/92	

Solutions

Sample#	Dil.Factor	1st Na+	2nd Na+	3rd Na+	4th Na+	Avg. Na
1	1/10			1120	1120	11200
2	1/10			1070	1080	10750
3	1/10			1040	1050	10450
4	1/10			990	998	9940
5	1/10			904	906	9050
6	1/10			843	845	8440
7	1/10	776	773			7745
8	1/10	711	712			7115
9	1/10	579	578			5785
10	1/10	468	471			4695
11	1/10	373	377			3750
12	1/10	270	272			2710
13	1/10	202	204			2030
14	1/10	169	163			1660
15	1/10	108	106			1070
16	1/10	73.3	73.6			734.5
17	1/10	*48.7	50.2			502
18	1/10	*24.3	28.4			284
19	1/10	*4.44	9.85			98.5
Slope		56.2	55.3	59.4	58.6	
Date		12/8/92	12/8/92	12/9/92	12/9/92	

Notes:

*: The lowest standard used was 50 ppm Na+ and measurements for samples 17-19 were extrapolated, therefore, use only the measurements provided by the 2nd calibration which includes a 10 ppm standard.

1 Dec 92
PD

0900

Data analysis of K^+ values associated with ISE measurements of K-Ca-Cl 0.05 N experimental solutions reveals inconsistencies. Based on calculated k^+ concentrations of both reference and experimental solutions and a phone conversation w/ T.G. concerning ISE reliability*, I feel it is necessary to re sample the experimental solutions. Data concerning final k^+ concentration will then be determined using flame-AAS using the same wavelength and linear range used for reference solution analysis. (SEE PG 245).

* of measurements cited on p. 242

- All experimental solutions will be resampled for K analysis
- Reference and exp. solutions may need to be resampled for Ca^{2+} analysis.

Overview:

Calculated and measured k^+ concentration values will be compared. An appropriate dilution factor (based on AAS linear range up to 20 ppm) will be selected. To minimize withdrawal of exp. solution volume, samples will be taken in 1 ml aliquots. La, as $LaCl_3 \cdot 7H_2O$ will be added to all samples during dilution. Sample dilutions appropriate for Ca^{2+} analysis will be ~~st~~ split for later (potential) Ca^{2+} analysis. Note: La is an appropriate chem. interference inhibitor for Ca.

Diluted samples will be analyzed for k^+ using a wave length setting which provides a linear range of up to 20 ppm K^+.

31 Dec 92
PD

K-Ca-Cl 0.05 N

Solution #	calculated init K	measured (AA) init K	calc. final K (isotherm)	meas. final K (ISE)	dilution factor
1	1759.2	1769	1913.5	1730	1/100
2	1759.2	1769	1875.2	1710	
3	1759.2	1769	1846.4	1670	
4	1759.2	1769	1804.	1640	
5	1563.6	1564.5	1754.1	1456.7	1/100
6	1563.6	1564.5	1665.4	1466.7	1/200
7	1368.3	1372.5	1559.2	1270	
8	1368.3	1372.5	1481	1262.5	
9	1172.8	1166	1362.6	1102	
10	1172.8	1166	1274.8	1130	
11	977.4	976	1138.6	934.5	1/200
12	781.9	766.5	942.46	756	1/100
13	586.4	570	783.2	618	1/100
14	391	375.5	544.4	423.7	1/50
15	195.5	194.75	433.4	263.3	1/50
16	195.5	194.75	346.6	243.7	1/50
17	195.5	194.75	243	215.3	1/25
18	0	0	105.8	58.9	1/10
19	0	0	66.7	40.6	1/10

SAME FOR Ca^{2+}

based on actual recorded wt. KCl p. 232

based on avg. AAS meas. p 245

KCA05.SPS printout

based on meas. ISE avg. p. 242

∴ dilution factors are appropriate for range of values, both calculated and ISE measurements.

31 Dec 92
PD
1045

0.1 % La, as $LaCl_3$, is the recommended (P-E) ionization interference inhibitor for K measurements using AAS. It is easiest to add La in solution form rather than solid form. A 1% La solution can be added to samples during the dilution process to give a final La concentration of 0.1%.

1% La solution (wt/vol) consists of $\frac{1\text{ g La}}{100\text{ ml solution}} = 0.01\text{ g/ml}$

For 1000 ml, 10 g La gives 0.01 g/ml

$10\text{ g La} \cdot \frac{371.38\text{ g } LaCl_3 \cdot 7H_2O}{138.9055\text{ g La}} = 26.7362\text{ g } LaCl_3 \cdot 7H_2O$ required to make 1% La solution.

bkr + $LaCl_3$ 130.5801 g
wt 250 ml bkr 102.7480 g

wt $LaCl_3 \cdot 7H_2O$ 27.8321 g - Fisher lot # 915032A

Add nanopure H_2O to 250 ml beaker, dissolve using teflon stir bar and mag. stirrer for mixing. Transfer solution to 1000 ml vol flask, include washings from beaker. Make up to mark, transfer to labeled 1 L poly bottle.

$27.8321\text{ g } LaCl_3 \cdot 7H_2O \cdot \frac{138.9055\text{ g La}}{371.38\text{ g } LaCl_3 \cdot 7H_2O} = 10.4094\text{ g La}$

$\frac{10.4094\text{ g La}}{1000\text{ ml}} \Rightarrow$ 1.04 % La solution

31 Dec 92
PD
1100

Sample and dilute K-Ca-Cl 0.05 N experimental solutions in preparation for AAS analysis.

① Swirl each solution for 30 sec (by hand), allow to sit for ≥ least 30 min so that fine zeolite material will settle out of solution.

② Sample experimental solutions (FOR EACH SOLUTION)

a. withdraw 1ml from ~~each~~ PD solution using volumetric pipette 12/31/92

b. discharge pipette into vol. flask sized as appropriate for dilution listed on p. 265. Wash flask neck w/ n H_2O.

c. Add 1.0% La solution to flask in amount so that 0.1% La solution will result after final dilution.

e.g. 10 ml of 1.0% La added to 100 ml vol flask will give 0.1% solution.

d. Add n H_2O to vol flask to just below the mark, mix well. Make up to mark w/ n H_2O, mix again.

e. Transfer sample dilution to clean, labeled PP bottle.

③ Record sample information on table p. 268.

- wt of exp solution bottle is included to provide solution volume information if needed for reverse isotherm experimental design.

31 Dec 92
PD

1310

solution #	volume removed (ml)	La (1.0%) added (ml)	diluted to (ml)	sol. wt before (g)	sol wt after (g)
1	1	10	100	22.3021	21.2622
2	1	10	100	22.1021	21.0707
3	1	10	100	21.8810	20.8512
4	1	10	100	50.5334	49.4892
5	1	10	100	22.1246	21.0885
6	1	20	200	50.6481	49.6070
7	0.5	10	100	21.8545	21.3254
8	1	20	200	50.8311	49.7919
9	0.5	10	100	21.0047	20.4749
10	1	20	200	50.5369	49.5024
11	0.5 (1)	10	100	50.3875	49.3221
12	1	10	100	50.4153	49.3671
13	1	10	100	41.5194	40.4824
14	1	5	50	41.3879	40.3551
15	1	5	50	41.2143	40.1693 10/31/92
16	1	5	50	50.5285	49.~~4869~~ PD 2189
17	1	2.5	25	108.1980	107.1809
18	1	1	10	107.5529	106.5153
19	1	1	10	108.0838	107.0417

1600

tal of 1ml → moved first .5ml was incorrectly diluted

↓ values approx. used graduated pipette

Sampling completed @ 1545

* Samples 6-11 split after dilution for future Ca^{2+} analysis.

31 Dec 92
PD

1610

- Prepared standard solutions for K AAS analysis
- Since all dilutions bring samples w/in the linear range of the method, a linear calibration using 4 standards and one check standard will be performed.

Linear range up to 20 ppm
check std as listed by Perkin-Elmer 4 ppm (to give 0.200 Abs)

standards to be made @ 5, 10, 15, 20 ppm
check @ 4 ppm

standards made by diluting purchased stock reference ~~sol'n~~ PD solution for K.

1000 ppm Fisher lot # 910846-18 exp. 2/93

$$20\text{ ppm} = \frac{2\text{ ml} \cdot 1000\text{ ppm}}{100\text{ ml}} \qquad 4\text{ ppm} = \frac{10\text{ ml} \times 20\text{ ppm}}{25\text{ ml}}$$ 12/31/92 PD

$$15\text{ ppm} = \frac{3\text{ ml} \cdot 1000\text{ ppm}}{200\text{ ml}} \qquad = \frac{20\text{ ml} \times 20\text{ ppm}}{50\text{ ml}}$$

$$10\text{ ppm} = \frac{1\text{ ml} \cdot 1000\text{ ppm}}{100\text{ ml}} \qquad 5\text{ ppm} = \frac{50\text{ ml} \times 10\text{ ppm}}{100\text{ ml}}$$

1.0% La solution added to all standards to give 0.1% final La concentration.

20 ppm → 10 ml La added
15 ppm → 20 ml La added
10 ppm → 10 ml La added

5 ppm → 5 ml La added
4 ppm → 4 ml La added

21 Dec 92 PB 1645

machine (AAS) set-up IAW P-E manual for K using linear range of 0-20 ppm.

- flame, Al/z had much drift => adjusted nebulizer inflow

- acetelyne tank press low

→ time and tank press required shutdown, samples will be run Monday 4 Jan 93.

4 Jan 93 PB 1100

- replaced acetylene tank for AAS PB PB 1/4/93 PB

- Completed, test sat.

1300

Set up flame AAS for K sample run.

blank, all standards and exp. solutions contained 0.1% La as $LaCl_3 \cdot 7H_2O$ in solution to minimize ionization interferences.

Based on information from past K analyses (see p. 245), the machine was optimized for the 0.20 ppm linear range.

Set-up summary:

Lamp - hollow cathode K, current: 6 ma

λ = 769.9 nm linear range 0.20 ppm

air-acetylene ← lean blue flame, some slight yellow color due to La in blank. (color not related to flame conditions)

acetylene 13 ~ 1.75 units @ 13 # air 5 units @ 57 #

4 Jan 93 Parameter entry: current: 6 ma
integration time: 2.0 sec
replicates: 5
calib: linear (2)
mode: flame 1/4/93
std 1 - ~~5.00~~ PB 5.00 ppm
2 - 10.00 ppm
3 - 15.00 ppm
4 - 20.00 ppm
no reslope

Al/z and 4 ppm sensitivity check run after 7, 14 and 19 samples

solution	ppm (avg. 5 reps)	std. dev.	solution	ppm	std. dev.
blank	0.00	0.0014			
5 ppm cal	5.00	.0013	14	8.68	.1641
10 ppm cal	1/4/93 ~~10.00~~ 10.01	.1144	15	5.39	.0275
15 ppm cal	1/4/93 ~~15.00~~ 14.94	.1049	16	4.86	.0260
20 ppm cal	1/4/93 ~~20.00~~ 19.86	.3600	17	8.50	.0350
4 ppm ck	4.10	.0412	18	6.19	.0646
PB 1	17.64	.1468	19	4.31	.1539 ~~.0411~~ 13 1/4/93
1/4/93 2	17.91	.1986	4 ppm ck (2)	4.08	.0256
3	1/4/93 PB ~~18.02~~ 17.91	.1175	4 ppm ck (3)	4.22	.1439
4	1/4/93 PB ~~16.37~~ 18.02	.1458	4 ppm ck (4)	4.12	.0207
5	1/4/93 PB ~~8.18~~ 16.37	.2082	10 ppm cal final	10.11	
6	1/4/93 PB ~~7.55~~ 8.18	.0618			
7	1/4/93 PB ~~7.14~~ 7.55	.0598			
8	1/4/93 PB 7.14	.0649			
9	6.31	.0422			
10	1/4/93 PB ~~5.27~~ 6.31	0.0549			
11	5.27	.1713			
12	8.36	.1618			
13	6.38	.1606			

1355

4 Jan 93
PB

K AAS data corrected for dilutions

exp. sol	AA (ppm)	dil	corrected (ppm)
1	17.64	1/100	1764
2	17.91	↑	1791
3	17.91		1791
4	18.02	↓	1802
5	16.37	1/100	1637
6	8.18	1/200	1636
7	7.55	↑	1510
8	7.14		1428
9	6.31		1262
10	6.31	↓	1262
11	5.27	1/200	1054
12	8.36	1/100	836
13	6.38	1/100	638
14	8.68	1/50	434
15	5.39	1/50	269.5
16	4.86	1/50	243
17	8.50	1/25	212.5
18	6.19	1/10	61.9
19	4.31	1/10	43.1

* (26 Jan 93) - 2nd analysis entered beginning p. 296

7 Jan 93
PB
1130

Sampled 0.5N $NaCl$-$SrCl_2 \cdot 7H_2O$ solutions (non-spiked references) ~~for~~ to make LSA blanks. 1/7/93

- Dispensed 10 ml of each ESr; solution into a 25 ml plastic LSA vial.
- weighed each vial before and after dispensing solution so that wt. of solution ~~is~~ can be found if needed. 1/7/93

7 Jan 93
PB

Non-spiked Esr reference solutions for use as LSA blanks (0.5N $NaCl$-$SrCl_2 \cdot 7H_2O$

solution	volume	weight (g) vial + solution	weight (g) vial empty	net weight (g) solution
Esr 0.05	10 ml	17.1173	6.9072	10.2101
0.1	10 ml	17.0614	6.8831	10.1783
0.2	10 ml	17.2397	7.0034	10.2363
0.3	10 ml	17.2388	6.9886	10.2502
0.4	10 ml	17.1296	6.8732	10.2564
0.5	10 ml	17.1700	6.9004	10.2696
0.6	10 ml	17.2424	6.9616	10.2808
0.7	10 ml	17.2744	6.9876	10.2868
0.8	10 ml	17.2107	6.9084	10.3023
0.9	10 ml	17.1939	6.8682	10.3257
1.0	10 ml	17.2374	6.8910	10.3464

Ca analysis

7 Jan 93
PB
1345

Resampled experimental solutions for KCl-$CaCl_2 \cdot 2H_2O$ isotherm experiment (0.05N) in preparation for AAS. Procedure LAW that outlined on p. 267. PB 1/7/93

solution #	vol. removed (ml)	La (0.00%) added (ml)	diluted to (ml)	sol. wt. before (g)	sol wt after (g)
1	1	2.5	25	21.2525	20.2286
2	1	2.5	25	21.0471	20.0169
3	1	2.5	25	20.8499	19.8154
4	1	2.5	25	49.4878	48.4449
5	1	2.6	25	21.0819	20.0504
* p. 274 → 12	1	25	250	49.3464	48.3553
13	1	25	250	40.4797	39.4403
14	1	25	250	40.3525	39.3156
15	1	25	250	40.1396	39.1138
16	1	25	250	49.2170	48.1842
17	1	25	250	107.1770	106.1506
18	1	25	250	106.4878	105.4489
19	1	25	250	107.0244	104.7735

7 Jan 93 Pp

* exp. solutions 6-11 sampled previously during K analysis preparation.
- samples are diluted to give an expected final concentration of Ca^{2+} within the 0-5 ppm range.

7 Jan 93 Pp

Prepared standards for Ca AAS analysis. The expected linear range for the wavelength to be used is 0-5 ppm Ca. Samples have been diluted to fall w/in this range. Using the linear calibration method on the P-E 3100 will require at least 3 standards. I select 1, 2, 4, and 5 ppm standard values. This will bracket the linear range quite well. ~~and~~ 1/13/93 The 4 ppm standard also serves as the sensitivity check standard for Ca analysis.

Using a purchased Ca reference solution containing 1000 ppm Ca, [Fisher lot #: 913291-24 exp 6/93] the 4 and 5 ppm standards will be diluted directly and the 1 and 2 ppm standards diluted serially from the 5 and 4 ppm stds respectively.

SUMMARY TABLE FOR STD PREP:

Standard	x (ml)	of (sol)(ppm)	diluted to	1.0% La added*
5 ppm	1 ml	1000	200 ml	20 ml
4 ppm	1 ml	1000	250 ml	25 ml
2 ppm	25 ml	4	50 ml	2.5 ml
1 ppm	20 ml	5	100 ml	8.0 ml

standards were transferred to labeled PP bottles immediately after final dilution and mixing. Bottles were capped tightly

* La (1.0%) added prior to final dilution

8 Jan 93 Pp

Set up flame AAS (P-E 3100) for Ca^{2+} analysis.

- all blanks, standards, and sample unknowns contained 0.1% La solution.
- machine set-up using parameters outlined in P-E manual Ca AAS worksheet.

SET-UP SUMMARY:

Lamp: hollow-cathode, Ca-Mg current: 5 ma
λ: 422.7 nm
flame: air-acetylene, lean, blue
linear range (expected):
slit: 0.7

air press: 57 # flow: 5 units
C_2H_2 press: 13 # flow: 1.75 units

PARAMETER ENTRY:

Lamp. current: 5 ma
Int. time: 2 sec
replicates: 5
calibration: linear (2)
method: flame
std 1: 1.00 ppm
std 2: 2.00 ppm
std 3: 4.00 ppm
std 4: 5.00 ppm
reslope: none

3 Jan 93
PB

Analysis worksheet

	Solution	Conc (ppm)	σ	Solution	Conc (ppm)	σ
	blank	0.00	0.0003	12	2.20	~~0.0148~~ 0.0199
	1 ppm	1.00	0.0004	13	2.58	~~0.0139~~ 0.0148
calibration	2 ppm	1.99	0.0103	14	2.88	~~0.0028~~ 0.0139
calibration	4 ppm	3.95	0.0088	blank (A/Z)	0.00	0.0028
calibration	5 ppm	4.90	0.0171	4 ppm	3.82	0.0214
	1	3.62	0.0231	15	3.19	0.0169
	2	3.73	0.0227	16	3.12	0.0164
	3	3.75	0.0179	17	3.28	0.0032
	4	3.81	0.0311	18	3.52	0.0212
	5	7.18	0.0447	19	3.60	0.0112
	6	0.98	0.0062	blank	-0.02	0.0037
	7	1.46	0.0044	4 ppm	3.84	0.0200
	blank (A/Z)	0.00	0.0025	2 ppm	1.95	0.0077
	4 ppm	3.82	0.0225	1 ppm	0.98	0.0057
	8	1.36	0.0086	5 ppm	4.70	0.0250
	9	1.86	0.0089			
	10	1.86	0.0067			
	11	2.37	0.0096			

(A/Z) - adjust zero
- flame adjusted using 4 ppm std.

The above results show a significant amount of drift from calibration to final sample. This drift is not corrected by A/Z and is evidenced by the 4ppm and 5 ppm readings at the end of the run.

Because dilution factors are high (up to 250x), I need better control of the output of the machine. I will run the samples again using the same machine set-up and dilutions.

8 Jan 93
PB

Exceptions for the second run will include:
- no machine calibration: calibration curve will be fitted manually using abs. values.
- values will be read in continuous absorbance mode

Obtaining the data in continuous mode (2 sec integration time) will provide visual information on Abs variance and will not allow the machine to manipulate the raw data.
-Five replicates will be run.

	Solution	Abs	Avg	ppm	Solution	Abs	Avg	ppm
*	blank (A/Z)	0.00	0.00	0	7	068, 069, 068 068, 068	0.068	1.48
	1 ppm	0.046, .047, .046 .047, .046	N/A	1.00	8	066, 066, 067 067, 066	0.066	1.44
	2 ppm	0.092, .092, .093 .092 .092	N/A	2.00	blank	0.00		
					4 ppm	0.179		
PB	4 ppm	0.180, .180, .181 .181 .180	N/A	4.00	9	088, 089, 089 089, 089	0.089	1.96
	5 ppm	0.223, .221, .224 .223, .221	N/A	5.00	10	088, 088, 089 088, 088	0.088	1.94
	1	.164, ~~.168~~ .163 .163, .164, .164	0.164	3.65	11	112, 111, 111 112, 113	0.112	2.48
	2	168 168 171 170 170	0.170	3.77	12	104, 104, 105 104, 104	0.104	2.30
	3	172, 173, 173 174, 172	0.173	3.86	13	122, 123, 122 123, 122	0.122	2.71
	4	175, 177, 176 176, 176	0.176	3.93	14	137, 138, 138 138, 137	0.138	3.07
	5	332, 332, 330 331 330	0.331 ~~0.332~~ 1/8/93	7.43	15	152, 152, 151 152, 151	0.152	3.38
	6	046, 045, 046 045 045	0.045	0.96	16	149, 150, 148 149, 148	0.149	3.32

Jan 93

pp

solution	Abs	Avg	ppm
17	0.154, 155, 154 153 153	0.152	3.38
18	164, 165, 165 163, 164	0.164	3.65
19	168, 167, 169 168, 169	0.168	3.74
4ppm	179, 178, 178 176, 177	0.178	3.97

pp

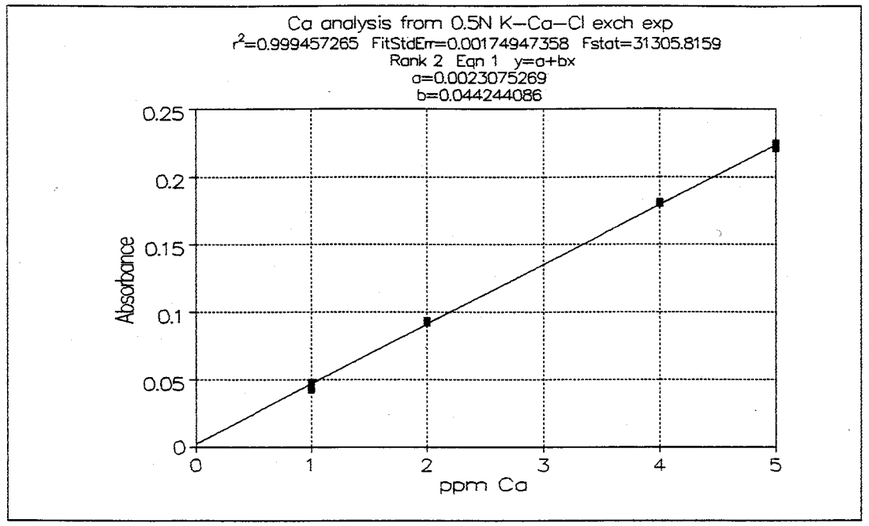
-results are much better, drift is ~~minimized~~ minimized. 1/8/93

-standard curve is fit using TBLCURVE 3.0, resulting equation, $y = mx + b$, is used to generate ppm values for unknowns. All standard data is used to generate fit => avg are N/A.

graph of standard fit and table of parameter values and error are shown on following page.

* - corrected results on page 286

Ca^{2+} calibration curve



Ca analysis from 0.5N K-Ca-Cl exch exp Jan 11,1993 10:06 AM
19 Active X-Y Points
X: ppm Ca Mean: 3.0526315789 SD: 1.649029486
Y: Absorbance Mean: 0.1373684211 SD: 0.0729796094
File Source: CAPPM05N.PRN

Rank 2 Eqn 1 y=a+bx

r2 Coef Det	DF Adj r2	Fit Std Err	F-value
0.9994572646	0.9993894227	0.0017494736	31305.815864

Parm	Value	Std Error	t-value	95% Confidence Limits	
a	0.002307527	0.000862423	2.675633564	0.000485674	0.00412938
b	0.044244086	0.000250059	176.934496	0.04371584	0.044772332

8 Jan 93 PB

subsampled non-spiked Na-Sr-Cl 0.5N reference solutions for use as blanks in LSA counting. Withdraw 10 ml from each solution $ESr_{0.05}$ - $ESr_{1.0}$. Placed solutions into 25 ml plastic LSA vials. Labeled each vial based on solution it contained (e.g. $ESr_{0.05}$) SEE P. 273

11 Jan 93 PB

K-Ca-Cl isotherm experiment

- Dilution of 0.05N reference solutions for analysis of Ca by flame AAS.

- based on measured (ISE) and calculated (wt $CaCl_2 \cdot 2H_2O$) concentration values, solutions will be diluted 1/250.

① mix solutions by swirling by hand

② For each solution:

a. withdraw 1.0 ml using volumetric pipette. (Remember to rinse pipette ~~thru~~ thoroughly) PB 1/11/93

b. transfer 1.0 ml to 250 ml vol flask. Rinse needle with nH_2O.

c. Add 1.0 % La to make 0.1% when diluted. (in this case ≈ 25 ml)

d. rinse needle and dilute to mark w/ nH_2O. mix well and transfer to labeled polypropylene bottle

③ analyze using AAS.

11 Jan 93 PB

Dilution table K-Ca-Cl 0.05N references

solution	vol sampled	La (1.0%) added	diluted to
~~1/11/93 PB ESr 0.05~~	~~1 ml~~	~~25 ml~~	~~250 ml~~
Xca 0.1	1 ml	25 ml	250 ml
0.2	1 ml	25 ml	
0.3	1 ml	25 ml	
0.4	1 ml	25 ml	
0.5	1 ml	[illegible] ml	
0.6	1 ml	[illegible] ml	
0.7	1 ml	25 ml	
0.8	1 ml	25 ml	
0.9	1 ml	25 ml	
1.0	1 ml	25 ml	

11 Jan 93 PB

Analysis of Ca in 0.05 K-Ca-Cl reference solutions (included are Bret Leslie's soil/rock sample solutions see notebook AN-1, CNWRA cont no. 024) p. 220-231

Machine (P-E 3100) setup summary:

- Wavelength, lamp, flame, sens check and linear range as prescribed by standard conditions sheet for Ca in P-E manual.

Lamp: Ca-Mg hollow cathode current: 5 ma
λ: 422.7 nm
flame: air-acetylene air: press - 56 # flow - 5.5
lean, blue C_2H_2: press - 13.2 # flow - 1.75

slit: 0.7 nm | high

sensitivity check: 4 ppm → flame adjusted to ~~1.96~~ 0.196 Abs 1/11/93 PB

1 Jan 93
PB

Ca analysis by AAS (cont'd)

Parameters:

current - 5 ma cal method - linear
int time - 2 seconds anal mode - flame
reps - 5 reslope std - none
stds 1: 1.00 ppm 3: 4.00 ppm
2: 2.00 ppm 4: 5.00 ppm

Interference additives: 0.1% La [as 1.0% La in $LaCl_3 \cdot 7H_2O$ solution] added to all stds, blanks, and unknowns. K^+ not added due to solution composition.

Other: all data obtained using continuous absorbance mode - machine calibration not used.

CALIBRATION: (A/z) w/ blank to 0.000 Abs units

standard no.	value	Abs
1	1 ppm	0.048, 0.048, 0.047, 0.048, 0.048
2	2 ppm	0.096, 0.098, 0.097, 0.097, 0.097
3	4 ppm	0.190, 0.190, 0.190, 0.0190, 0.188
4	5 ppm	0.236, 0.234, 0.234, 0.236, 0.237

all abs values used to fit calibration curve

11 Jan 93
PB

UNKNOWN SAMPLES:

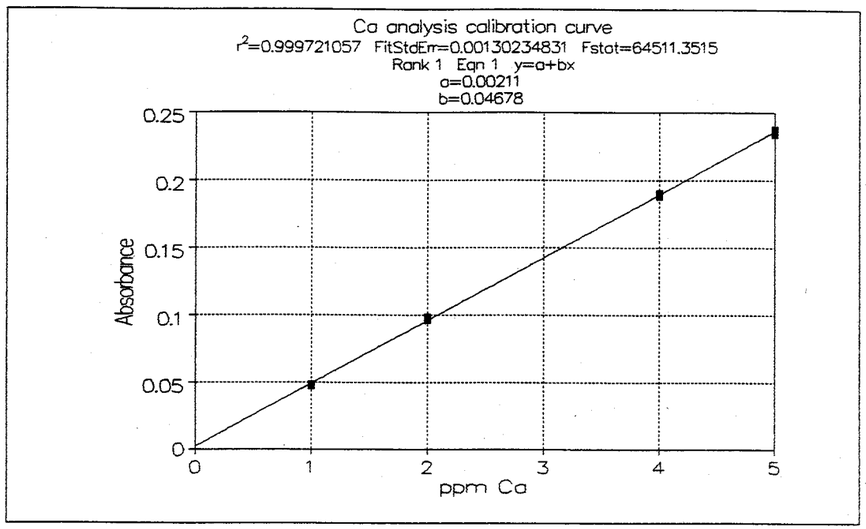
	Solution	Abs	Avg.	cal ppm	Abs σ(s)
	XCa 0.1	0.018, 0.018, 0.017, 0.017, 0.016	0.017	0.32	0.0008
	XCa 1.0	0.184, 0.184, 0.184, 0.184, 0.185	0.184	3.89	0.0004
	XCa 0.8	0.146, 0.146, 0.147, 0.146, 0.147	0.146	3.08	0.0005
	XCa 0.6	0.113, 0.114, 0.114, 0.114, 0.113	0.114	2.39	0.0005
	XCa 0.2	0.037, 0.038, 0.037, 0.037, 0.038	0.037	0.75	0.0005
	XCa 0.3	0.049, 0.050, 0.050, 0.050, 0.050	0.050	1.02	0.0004
	4 ppm	0.191, 0.191, 0.192, 0.191, 0.192	0.191	4.04	0.0005
	blank	(A/z) 0.000	—	—	—
	XCa 0.4	0.075, 0.075, 0.074, 0.075, 0.075	0.075	1.56	0.0004
	XCa 0.5	0.093, 0.093, 0.093, 0.093, 0.093	0.093	1.94	0.0000
	XCa 0.9	0.165, 0.168, 0.166, 0.167, 0.167	0.167	3.52	0.0011
	XCa 0.7	0.126, 0.126, 0.127, 0.126, 0.126	0.126	2.65	0.0004
	4 ppm	0.191, 0.190, 0.190, 0.192, 0.191	0.191	4.04	0.0008
	2 ppm	0.098, 0.098, 0.097, 0.098, 0.098	0.098	2.05	0.0004
	5 ppm	0.235, 0.235, 0.236, 0.238, 0.235	0.236	5.0	0.0013
	blank	(A/z) 0.000	—	—	—
Above max *	5-A*C*Ca	~~0.0~~ 1/11/93 PB 0.560, 0.559, 0.558	0.559	11.9	0.0010
	5-B*C*Ca	0.155, 0.156, 0.156, 0.156, 0.158	0.156	3.29	0.0011
	5-C*C*Ca	0.030, 0.030, 0.030, 0.030, 0.031	0.030	0.6	0.0004
	7-C*C*Ca	0.012, 0.012, 0.012, 0.012, 0.012	0.012	0.21	0.0000
	7-B*C*Ca	0.068, 0.068, 0.068, 0.068, 0.068	0.068	1.41	0.0000
Above max *	7-A*C*Ca	0.250, 0.251, 0.251, 0.251, 0.249	0.250	5.3	0.0009
	8-A*C*Ca	0.033, 0.033, 0.033, 0.033, 0.033	0.033	0.66	0.0000
	blank	0.000, 0.000, 0.000, 0.000	0.000	0	0.0000
	4 ppm	0.191, 0.191, 0.190, 0.190, 0.192	0.191	4.04	0.0008
	8-B*C*Ca	0.008, 0.008, 0.007, 0.008, 0.007	0.008	0.12	0.0005
below MDL *	8-C*C*Ca	0.001, 0.000, 0.001, 0.001, 0.000	0.001	* -0.02	0.0005
Above max *	9-A*C*Ca	0.549, 0.553, 0.552, 0.549, 0.550	0.551	11.7	0.0018
	9-B*C*Ca	0.148, 0.148, 0.148, 0.148, 0.147	0.148	3.12	0.0004
	9-C*C*Ca	0.032, 0.031, 0.031, 0.031, 0.031	0.031	0.62	0.0004
	B1*C*Ca	0.000, 0.000, 0.000, 0.000, 0.000	0.000	0	0.0000

Jan 93
PB

UNKNOWN SAMPLES (cont'd)

solution	Abs	Avg	cal ppm	Abs S
blank	0.000, 0.000, 0.000, 0.000, 0.000	0.000	0	0.0000
4ppm	0.190, 0.190, 0.191, 0.191, 0.191	0.191	4.04	0.0005
2ppm	0.098, 0.098, 0.099, 0.098, 0.098	0.098	2.05	0.0004

All cal ppm values derived from calibration curve shown below.




```
Ca analysis calibration curve                    Jan 11,1993 1:56 PM
20 Active X-Y Points
X: ppm Ca                 Mean: 3                SD: 1.6222142113
Y: Absorbance             Mean: 0.14245          SD: 0.0758977671
File Source: CAREF.PRN

Rank 1  Eqn 1  y=a+bx

r2 Coef Det    DF Adj r2      Fit Std Err    F-value
0.9997210572   0.9996882404   0.0013023483   64511.351458

 Parm  Value        Std Error    t-value      95% Confidence Limits
 a     0.00211      0.000624584  3.378246906  0.000796554  0.003423446
 b     0.04678      0.00018418   253.9908492  0.046392686  0.047167314
```


11 Jan 93
PB

calibration fits from 8 Jan 93 and 11 Jan 93 are in excellent agreement [same stds used]. ~~Mim~~ 1/11/93 calculated minimum detectable calcium is ~ 0.08 ppm. This corresponds well with sens. value of 0.09 listed on standard condition sheet.

Ca-Mg lamp is very stable for Ca. QA checks show drift of 0.001 or less.

12 Jan 93
PB

AAS values for Ca in K-Ca-Cl 0.05N reference solutions

reference	cal ppm	dil factor	corr. ppm
Xca 0.1	0.32	250	80.0
0.2	0.75	250	~~187.5~~ 188
0.3	1.02	250	255.0
0.4	1.56	250	390.0
0.5	1.94	250	485
0.6	2.39	250	598
0.7	2.65	250	663
0.8	3.08	250	770
0.9	3.52	250	880
1.0	3.89	250	973

Corrections for samples 5-A*C*Ca through 9-C*C*Ca are shown in notebook AN-1, CNWRA cont. no. 024 p. 228-231.

12 Jan 93
PP

AAS values for Ca in K-Ca-Cl 0.05 N exp. solutions
from p. 277-278.

exp solution	cal ppm	dil factor	corrected ppm
1	3.65	25	91
2	3.77	25	94
3	3.86	25	97
4	3.93	25	98
5	7.43	25	186
6	0.96	200	192
7	1.48	200	296
8	1.44	200	288
9	1.96	200	392
10	1.94	200	388
11	2.48	200	496
12	2.30	250	575
13	2.71	250	678
14	3.07	250	768 1/12/93
15	3.38	250	~~845~~ PP 845
16	3.32	250	~~4~~ PP 830
17	3.38	250	845
18	3.65	250	913
19	3.74	250	935

13 Jan 93
PP

Na - ^{90}Sr - Cl 0.5 N isotherm experiment

The spike solution used in the ^{90}Sr sorption exp. includes carrier Sr and Y in ~~conc~~ PP 1/13/93 concentrations of 100 ppm each. How does the conc. of carrier impact the concentration of Sr in the reference solutions?

Assuming 200 mg ~~weight~~ PP 1/13/93 occupies 200 µl
and knowing that 1 ppm ≈ 1 µg/ml

Each ref solution contains ~ 0.200 g ^{90}Sr spike

$(0.200\ ml)\ \frac{100\ \mu g}{ml} = 20\ \mu g$ Sr added per ref solution

Each ref solution contains ~ 200 g solvent

$\frac{20\ \mu g}{200\ g} = 0.1$ ppm added due to addition of spike

For 0.5N ESr solutions, the minimum concentration of Sr from $SrCl_2 \cdot 6H_2O$ is 1095 ppm ⇒ the spike addition contributes a maximum of 0.01 % of Sr content in solution ∴ it is reasonable that the increase in $[Sr^{2+}]$ due to spike addition be neglected.

Prepared 1% La solution for use as an interference inhibitor during AAS analysis.

26.7362 g $LaCl_3 \cdot 7H_2O$ required to make 1000 ml of 1.0% La solution.

$LaCl_3 \cdot 7H_2O$: Fisher lot # 915032A

blr + $LaCl_3$ (g)	124.0654
wt 250 ml blr (g)	97.3186
	26.7468

procedure as stated on p. 266

$26.7468\ g\ LaCl_3 \cdot 7H_2O \cdot \frac{138.9055\ g\ La}{371.38\ g\ LaCl_3 \cdot 7H_2O} = 10.0040\ g\ La$

$\frac{10.0040\ g\ La}{1000\ ml} = 1.0004\ \%$ La solution

4 Jan 93
PP

Resampled selected K-Ca-Cl 0.05N experimental and reference solutions for verification analysis of Ca using AAS.

An attempt was made to dilute solutions using different dilution factors (with larger initial withdrawal volumes) to check previous readings.

Sample withdrawal and dilution:

- After mixing 2 or 5 ml sample aliquots were withdrawn from reference or exp. solutions using volumetric pipettes.

- Pipette was discharged into vol flask of appropriate size (50 or 250 ml).

- 1.0% La was added in an amount to give 0.1% La solution after dilution of sample

- nH_2O added to flask, made up to mark

- Dilution was mixed and transferred to PP bottle.

* volumetric ~~ffsk~~ pipettes and PP bottles pre-washed with solution prior to use.

Summary Table

	solution	vol taken	La added	dil. to	wt before	wt after
exp	1	2 ml	5 ml	50 ml	20.2260 g	18.1978 g
	2	2 ml	5 ml	50 ml	19.9810 g	17.9464 g
	3	2 ml	5 ml	50 ml	19.8131 g	17.7815 g
	4	5 ml	25 ml	250 ml	48.4404 g	43.0628 g
ref	0.1	5 ml	25 ml	250 ml	–	–
	0.2	5 ml	25 ml	250 ml	–	–

14 Jan 93
PP

Performed AAS analysis for Ca on selected ~~K-Ca-Cl~~ K-Ca-Cl 0.05N exp. and ref. solutions

PE 3100 set up summary:

- λ, linear range, sens check, lamp and interferences as noted on Ca standard conditions sheet

Lamp: Ca-Mg hollow cathode — current: 5 ma
λ: 422.7 nm — lin range: 0-5 ppm
flame: air-acetylene — air: press: ~~13.5#~~ 56 flow: 5.5
lean, blue — C_2H_2: press 13.5# flow: 1.75
slit: 0.7 nm, high
sens. check 4.00 ppm adjusted → 0.209 Abs

Parameters:

current:	5 ma	cal method:	linear
Int. time:	2 sec	anal mode:	flame
reps:	5	rcslope:	none
stds	1- 1.00	3- 4.00	
	2- 2.00	4- 5.00	

Interference additives: 0.1% La added to all samples, blanks, and standards

Other: Machine calibration not used, readings and stds measured in continuous Abs mode.

4 Jun 93
PB

On AAs analysis (cont'd)

Calibration: Absorbance

blank (A/2)	
1.00 ppm	0.053, 0.053, 0.053, 0.054, 0.053
2.00 ppm	0.107, 0.107, 0.108, 0.107, 0.108
4.00 ppm	0.211, 0.210, 0.211, 0.211, 0.212
5.00 ppm	0.269, 0.271, 0.270, 0270, 0270

Unknowns:

sample	Abs	Avg	S	Cal ppm
exp 1	0.199, 0.199, 0.200, 0.199, 0.199	0.199	0.0004	3.72
2	0.205, 0.204, 0.205, 0.205, 0.205	0.205	0.0004	3.83
3	0.208, 0.208, 0.209, 0.208, 0.209	0.208	0.0004	3.89
4	0.106, 0.106, 0.107, 0.107, 0.108	0.107	0.0008	2.01
ref 0.1	0.109, 0.108, 0.109, 0.109, 0.109	0.109	0.0004	2.04
0.2	0.210, 0.211, 0.210, 0.211, 0.212	0.211	0.0008	3.94
4 ppm	0.217, 0.217, 0.215, 0.218, 0.217	0.217	0.0011	4.2
2 ppm	0.112, 0.112, 0.112, 0.112, 0.111	0.112	0.0004	2.1

(*) calibration curve and corrected values shown on pp. 292-298

Jan 93
PB

Na-Sr-Cl 0.5N isotherm experiment

Reference solution analysis for Sr using AAS.

Sr concentration can be verified by analyzing each ESr_x solution for Sr. The linear range for Sr using hollow-cathode lamp at 460.7 nm is 0-5 ppm. Since Sr concentrations are high, dilutions must be made serially.

15 Jun 93
PB

Sr AAs analysis (cont'd)

solution	calc. initial ppm	dilution 1	dilution 2	1.0% La added
$ESr_{0.05}$	1095	1/250	-	25
0.1	2190	1/500	-	50
0.2	4380	2/100	1/100	10
0.3	6570	2/100	1/100	10
0.4	8760	2/100	1/100	10
0.5	10950	2/100	1/100	10
0.6	13140	2/100	1/100	10
0.7	15330	2/100	1/100	10
0.8	17525	2/100	1/100	10
0.9	19710	2/100	1/100	10
1.0	21900	2/100	1/100	10

Sr is analyzed using nitrous oxide - acetylene

Sr standard dilutions

reference standard - 1000 ± 5 ppm
Ricca lot # S096 exp. 3/93

sensitivity check is 5.0 ppm
linear range 0-5 ppm

Standards of 1, 1.5, 2.5, and 5 ppm will be made

① 50 ppm stock solution

$$\frac{1000\ \text{ppm} \cdot 5\text{ml}}{100\ \text{ml}} = 50\ \text{ppm}$$

; Jan 93
PP

② Standards

a. $\frac{50\ ppm \cdot 10\ ml}{100\ ml} = 5\ ppm$ — 10 ml 1.0% La added

b. $\frac{50\ ppm \cdot 5\ ml}{100\ ml} = 2.5\ ppm$ — 10 ml 1.0% La added

c. $\frac{50\ ppm \cdot 3\ ml}{100\ ml} = 1.5\ ppm$ — 10 ml 1.0% La added

d. $\frac{50\ ppm \cdot 2ml}{100\ ml} = 1.0\ ppm$ — 10 ml La 1.0% added

Test for nitrous oxide - acetylene burner operation -

① removed air-acetylene burner

* locking ring was not attached properly!

② installed NO_2-C_2H_2 head

③ ignited flame air-acetylene then to NO_2-C_2H_2 following procedure in PE 3100 ops manual.

④ There was significant carbon accumulation when reducing conditions (6 flow units fuel 4 units oxidant) were created. Carbon xtls formed which would interfere ~~with~~ with readings.

⑤ NO_2 flame shutdown and burner replaced with air-acetylene burner.

* Sr can be analyzed using air-acetylene.

15 Jan 93
PP

Potential problems with analysis of Sr using an air-acetylene flame are the increased potential ionization interference effects.

PE 3100 set up summary:

→ , lamp, sensitivity check IAW PE AAS standard conditions sheet for Sr. Slit setting of 0.4 nm is not an option on the 3100, 0.7 nm will be used.

Lamp: Sr, hollow-cathode — current: 8 ma
λ: 460.7 nm — linear range: 0-5 ppm
flame: air-acetylene — air: press - 56# flow - 5.5
lean-blue — C_2H_2: press - 13.5# flow - 1.75
slit: 0.7 nm / high
sens. check 5.00 ppm adjusted → 0.191 Abs

Parameters:

current: 8 ma — cal method: linear
int. time: 2 sec — anal mode: flame
reps: 5 — reslope: none
stds: 1 - 1.00 — 3 - 2.50
2 - 1.50 — 4 - 5.00

Interference: 0.1% La added to all samples, stds, and blanks, Na present in all but 1.0 sample.

Other: machine calibration not used, readings made in continuous absorbance mode. Air-C_2H_2 flame used, slit @ 0.7nm.

Jan 93 Sr AAS analysis

Calibration:

~~blank~~

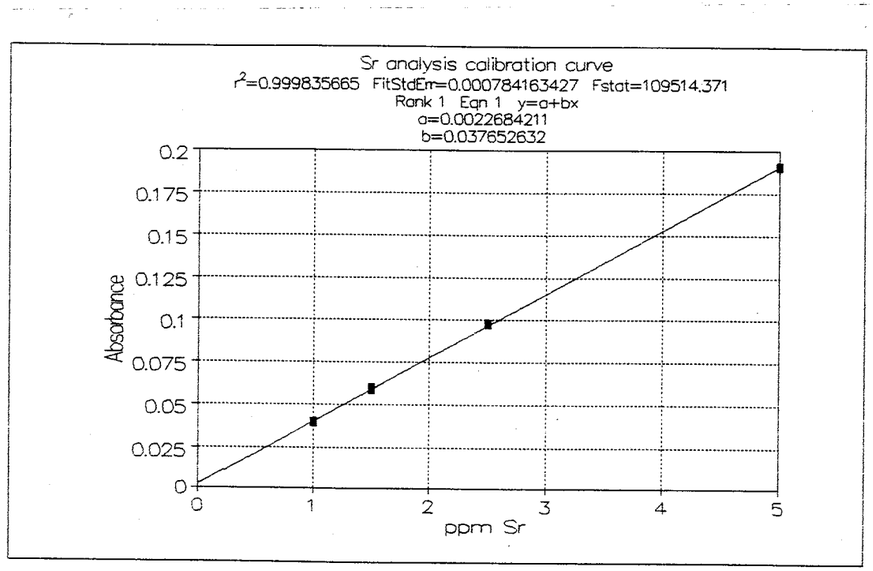
standard	Abs
blank (A/Z)	0.000
1.0 ppm	0.040, 0.039, 0.039, 0.039, 0.039
1.5 ppm	0.059, 0.059, 0.060, 0.059, 0.058
2.5 ppm	0.098, 0.097, 0.097, 0.097, 0.097
5.0 ppm	0.190, 0.190, 0.191, 0.190, 0.190

Samples:

solution	Abs	S	Avg	cal. ppm
ESr 0.05	0.175, 0.174, 0.175, 0.175, 0.174	0.0005	0.175	4.59
0.4	0.069, 0.069, 0.069, 0.068, 0.069	0.0004	0.069	1.77
0.3	0.053, 0.053, 0.053, 0.053, 0.053	0.0000	0.053	1.35
0.9	0.151, 0.151, 0.152, 0.152, 0.152	0.0005	0.152	3.98
0.2	0.035, 0.035, 0.035, 0.036, 0.035	0.0004	0.035	0.87
blank	0.000, 0.000, 0.000, 0.000, 0.000	0.000	0.000	
5 ppm	0.191, 0.192, 0.193, 0.191, 0.191	~~0.192~~ 0.0009 1/15/93	0.192	5.04
0.1	0.175, 0.176, 0.175, 0.175, 0.174	0.0007	0.175	4.59
0.5	0.088, 0.089, 0.088, 0.089, 0.088	0.0005	0.088	2.28
0.6	0.104, 0.103, 0.105, 0.105, 0.105	0.0009	0.104	2.70
0.8	0.138, 0.138, 0.139, 0.138, 0.139	0.0005	0.138	3.60
1.0	0.171, 0.170, 0.172, 0.170, 0.172	0.0010	0.171	4.48
0.7	0.122, 0.124, 0.122, 0.123, 0.122	0.0009	0.123	3.21
blank	0.000, 0.001, 0.001, 0.000, 0.000	0.0005	0.000	
5 ppm	0.191, 0.192, 0.192, 0.193, 0.193	0.0008	0.192	5.04
2.5 ppm	0.099, 0.099, 0.100, 0.100, 0.099	0.0005	0.099	2.57
1 ppm	0.041, 0.042, 0.041, 0.041, 0.041	0.0004	0.041	1.03

15 Jan 93 Sr readings are linear and stable. Na and La present inhibits most interference and solutions are simple (no competing cations) => air-acetylene flame useful for Sr.

Cal curve for Sr AAS analysis




```
Sr analysis calibration curve                    Jan 15.1993 5:21 PM
20 Active X-Y Points
X: ppm Sr                 Mean: 2.5              SD: 1.5811388301
Y: Absorbance             Mean: 0.0964           SD: 0.0595389302
File Source: SRPPMCAL.PRN

Rank 1  Eqn 1  y=a+bx

r2 Coef Det     DF Adj r2       Fit Std Err     F-value
0.999835665     0.9998163315    0.0007841634    109514.3709

 Parm  Value          Std Error     t-value       95% Confidence Limits
 a     0.002268421    0.000334148   6.788664386   0.001565736   0.002971106
 b     0.037652632    0.000113778   330.9295558   0.037413366   0.037891898
```

8 Jan 93 PD — Corrected Sr ppm values for Na-Sr-Cl 0.5N reference solutions

solution Sr	AAS cal ppm	dil factor	corr. ppm	% variance from calc ppm
0.05	4.59	1/250	1148	4.8
0.1	4.59	1/500	2295	~~4.8~~ PD 1/18/93 4.8
0.2	0.87	1/5000	4350	0.7
0.3	1.35	"	6750	2.7
0.4	1.77	"	8850	1.0
0.5	2.28	"	11400	4.1
0.6	2.70	"	13500	2.7
0.7	3.21	"	16050	4.7
0.8	3.60	"	18000	2.7
0.9	3.98	"	19900	1.0
1.0	4.48	"	22400	2.2

Avg 2.9 %

6 Jan 93 PD — I neglected to enter values/results from a second K analysis (using AAS) conducted 4 Jan 93. The following data are a continuation of that accumulated on 4 Jan 93. SEE PAGES 269-272.

Machine set-up as stated on pp. 270-271

* From 4 Jan 93, 1625

solution	calibration value	σ
blank	0	0.0069
5 ppm	5.00	0.0042
10 ppm	10.03	0.0903
15 ppm	15.05	0.2622
20 ppm	20.21	0.1414
4 ppm	3.77	0.0159

26 Jan 93 PD

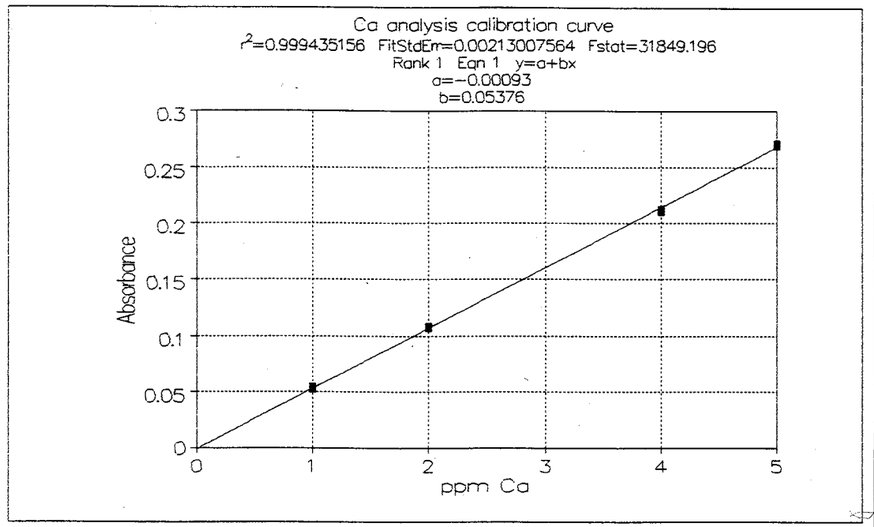
solution	ppm	corrected ppm
1	18.05	1805
2	17.85	1785
3	17.88	1788
4	18.00	1800
5	16.03	1603
6	8.05	1610
A/3	0	~~PD 148~~ 1/26/93 0
4 ppm	4.19	4.19
7	7.41	1482
8	7.10	1420
9	6.32	1264
10	6.19	1238
11	5.47	1094
12	8.36	836
13	6.41	641
14	8.72	~~PD 872~~ 436
15	5.54	~~PD 554~~ 277
16	4.93	~~PD 493~~ 247
17	8.42	~~PD 842~~ 211
18	6.21	621
19	4.35	43.5

26 Jan 93 PD — Corrected values from Cu analysis p. 290

sample	ppm	dil factor	corrected ppm
exp 1	3.72	1/25	93
exp 2	3.83	1/25	96
exp 3	3.89	1/25	97
exp 4	2.01	1/50	100.5
ref 0.1	2.04	1/50	102
ref 0.2	3.94	1/50	197

26 Jan 93
PD

calibration curve from 14 Jan 93 Ca analysis. see p. 290



Ca analysis calibration curve Jan 14,1993 11:55 AM
20 Active X-Y Points
X: ppm Ca Mean: 3 SD: 1.6222142113
Y: Absorbance Mean: 0.16035 SD: 0.0872348765
File Source: CAPPM2.PRN

Rank 1 Eqn 1 y=a+bx

r2 Coef Det	DF Adj r2	Fit Std Err	F-value
0.9994351558	0.9993687036	0.0021300756	31849.196033

Parm	Value	Std Error	t-value	95% Confidence Limits	
a	-0.00093	0.001021548	-0.91038272	-0.00307823	0.001218226
b	0.05376	0.000301238	178.4634305	0.053126523	0.054393477

27 Jan 93
PD

LSA results are posted on the ~~floppy~~ 1/27/93 following pages.

Spiked references and experimental solutions were counted. Also included were solutions spiked to measure/verify counting efficiency (for Cerenkov) and sample blanks. Methods for preparing solutions 9A-9G may be found in the radiochemistry notebook CNWRA controlled copy 031 pp. 160-162.

27 Jan 93

PB

Protocol #: 7 Name:CERENKOV Sr-90 10-Jan-93 11:53
Region A: LL-UL= 0.0-30.0 Lcr= 0 Bkg= 0.00 %2 Sigma=1.00
Region B: LL-UL=30.0-2000 Lcr= 0 Bkg= 0.00 %2 Sigma=0.50
Region C: LL-UL= 0.0-2000 Lcr= 0 Bkg= 0.00 %2 Sigma=0.10
Time =999.99 QIP = SIS
Sr-Na ion exchange experiment
High Sensitivity Count Mode

S#	TIME	CPMA	A:2S%	CPMB	CPMC	SIS	SOLUTION ~~FLAG~~
1	4.89	8193.46	1.00	33.95	8227.40	29.925	9A
2	9.69	4130.24	1.00	23.43	4153.66	30.344	9B
3	19.07	2098.11	1.00	14.58	2112.69	30.362	9C
4	49.04	815.82	1.00	10.83	826.65	32.030	9D
5	97.81	408.96	1.00	9.55	418.51	33.804	9E
6	197.29	202.76	1.00	7.96	210.72	37.274	9F
7	440.18	90.87	1.00	7.67	98.54	44.570	9G
(5 missing vials)							
13	309.13	129.40	1.00	7.95	137.35	40.751	1
14	60.05	666.14	1.00	10.47	676.62	32.972	2
15	67.81	589.99	1.00	10.62	600.62	33.114	3
16	80.50	496.96	1.00	9.24	506.20	33.420	4
17	49.05	815.58	1.00	11.78	827.36	32.731	5
18	56.77	704.60	1.00	11.36	715.96	32.920	6
19	59.79	669.08	1.00	10.70	679.78	33.064	7
20	50.95	785.22	1.00	11.85	797.08	32.715	8
21	48.99	816.49	1.00	11.21	827.70	32.715	9
22	49.25	812.26	1.00	11.33	823.59	32.809	10
23	49.67	805.36	1.00	11.80	817.15	32.662	11
24	49.89	801.84	1.00	11.59	813.45	32.691	12
25	4.79	8359.29	1.00	47.81	8407.10	31.384	0.05
26	4.79	8359.50	1.00	46.97	8406.47	31.070	0.1
27	4.78	8368.62	1.00	47.49	8416.11	31.156	0.2
28	4.78	8383.68	1.00	48.12	8431.59	31.255	0.3
29	4.81	8323.70	1.00	53.43	8377.13	31.298	0.4
30	4.78	8375.94	1.00	53.14	8429.08	31.423	0.5
31	4.84	8266.53	1.00	45.04	8311.57	31.398	0.6
32	4.69	8530.92	1.00	51.39	8582.30	31.369	0.7
33	4.79	8368.06	1.00	44.47	8412.74	31.099	0.8
34	4.80	8334.17	1.00	45.42	8379.58	31.002	0.9
35	4.76	8419.75	1.00	51.05	8470.80	31.039	1.0
(1 missing vial)							
37	52.52	761.82	1.00	10.55	772.37	32.687	13
38	49.33	811.03	1.00	12.14	823.17	33.023	14
39	52.17	766.80	1.00	11.48	778.30	32.724	15
40	51.25	780.53	1.00	11.69	792.23	32.875	16
41	49.52	807.88	1.00	11.21	819.08	32.565	17
42	48.19	830.09	1.00	11.75	841.81	33.055	18
43	47.65	839.52	1.00	11.71	851.23	32.888	19
(2 missing vials)							
46	999.98	16.84	1.54	2.38	19.22	54.420	BLANK
(2 missing vials)							
49	999.98	13.07	1.75	7.83	20.91	100.99	0.05B
50	999.98	13.88	1.70	7.71	21.58	97.235	0.1B
51	999.98	13.09	1.75	7.78	20.87	101.61	0.2B
52	999.98	13.54	1.72	7.93	21.47	100.45	0.3B
53	999.98	13.48	1.72	7.86	21.34	100.93	0.4B
54	999.98	13.42	1.73	7.97	21.39	101.22	0.5B
55	999.98	13.28	1.74	7.92	21.20	102.87	0.6B
56	999.98	13.42	1.73	8.12	21.54	102.27	0.7B
57	999.98	13.69	1.71	7.96	21.65	100.87	0.8B
58	999.98	13.44	1.73	8.07	21.51	102.93	0.9B
59	999.98	13.10	1.75	7.89	20.99	103.24	1.0B

SYSTEM NORMALIZED
C14 IPA DATA PROCESSED
C14 CHI SQUARE IPA DATA PROCESSED
H3 IPA DATA PROCESSED
H3 CHI SQUARE IPA DATA PROCESSED
BKG IPA DATA PROCESSED

27 Jan 93 PB

Protocol #: 7 Name:CERENKOV Sr-90 20-Jan-93 14:14
Region A: LL-UL= 0.0-30.0 Lcr= 0 Bkg= 0.00 %2 Sigma=1.00
Region B: LL-UL=30.0-2000 Lcr= 0 Bkg= 0.00 %2 Sigma=0.50
Region C: LL-UL= 0.0-2000 Lcr= 0 Bkg= 0.00 %2 Sigma=0.10
Time =999.99 QIP = SIS
Sr-Na ion exchange experiment
High Sensitivity Count Mode

S#	TIME	CPMA	A:2S%	CPMB	CPMC	SIS	SOLUTION ~~FLAG~~ PB
1	4.87	8223.00	1.00	35.52	8258.32	29.853	9A
2	9.71	4121.42	1.00	21.73	4143.15	30.212	9B
3	19.07	2098.16	1.00	14.37	2112.53	30.497	9C
4	49.30	811.36	1.00	10.20	821.56	32.010	9D
5	98.73	405.20	1.00	9.37	414.56	33.891	9E
6	195.16	204.96	1.00	8.54	213.50	37.315	9F
7	438.95	91.13	1.00	7.98	99.11	44.875	9G
(5 missing vials)							
13	312.20	128.14	1.00	8.36	136.49	40.699	EXP 1
14	60.65	659.59	1.00	10.31	669.88	32.693	EXP 2
15	68.33	585.41	1.00	10.52	595.93	32.931	EXP 3
16	80.32	498.08	1.00	9.65	507.74	33.164	EXP 4
17	49.34	810.76	1.00	11.53	822.29	32.417	EXP 5
18	57.85	691.46	1.00	10.25	701.73	32.460	EXP 6
19	60.38	662.50	1.00	10.62	673.12	32.659	EXP 7
20	51.08	783.12	1.00	10.69	793.81	32.385	EXP 8
21	49.13	814.21	1.00	11.24	825.46	32.291	EXP 9
22	49.27	811.91	1.00	10.84	822.77	32.446	EXP 10
23	50.07	799.02	1.00	10.74	809.77	32.016	EXP 11
24	50.38	794.05	1.00	11.14	805.20	32.505	EXP 12
25	4.82	8309.75	1.00	44.61	8354.36	30.916	0.05
26	4.83	8283.23	1.00	41.41	8324.64	30.896	0.1
27	4.81	8332.43	1.00	47.40	8379.83	30.914	0.2
28	4.77	8386.37	1.00	45.70	8432.08	30.862	0.3
29	4.82	8308.92	1.00	50.00	8358.92	30.869	0.4
30	4.83	8293.58	1.00	43.69	8337.06	30.964	0.5
31	4.77	8388.05	1.00	47.59	8435.64	30.929	0.6
32	4.72	8490.25	1.00	45.76	8536.02	30.925	0.7
33	4.73	8472.73	1.00	40.38	8512.90	30.818	0.8
34	4.77	8400.00	1.00	40.25	8440.25	30.638	0.9
35	4.80	8334.38	1.00	41.88	8376.25	30.562	1.0
(1 missing vial)							
37	52.58	760.88	1.00	11.43	772.31	32.499	EXP 13
38	48.91	801.50	1.00	10.70	812.20	32.119	EXP 14
39	52.67	759.58	1.00	10.48	770.06	32.386	EXP 15
40	50.98	784.86	1.00	10.57	795.43	32.336	EXP 16
41	49.89	801.82	1.00	11.41	813.23	32.465	EXP 17
42	48.81	819.63	1.00	11.35	830.98	32.422	EXP 18
43	47.55	841.45	1.00	11.71	853.19	32.543	EXP 19
(2 missing vials)							
46	999.98	16.90	1.54	2.30	19.20	53.592	BLANK

SYSTEM NORMALIZED
C14 IPA DATA PROCESSED
C14 CHI SQUARE IPA DATA PROCESSED
H3 IPA DATA PROCESSED
H3 CHI SQUARE IPA DATA PROCESSED
BKG IPA DATA PROCESSED

7 Jan 93
PB

```
Protocol #: 7        Name:CERENKOV Sr-90          22-Jan-93   21:47
Region A: LL-UL= 0.0-30.0  Lcr=   0  Bkg= 0.00  %2 Sigma=1.00
Region B: LL-UL=30.0-2000  Lcr=   0  Bkg= 0.00  %2 Sigma=0.50
Region C: LL-UL= 0.0-2000  Lcr=   0  Bkg= 0.00  %2 Sigma=0.10
Time =999.99      QIP = SIS
Sr-Na ion exchange experiment
High Sensitivity Count Mode
```

S#	TIME	CPMA	A:2S%	CPMB	CPMC	SIS	SOLUTION ~~FLAG~~ PB	
1	4.92	8140.65	1.00	33.54	8174.19	30.550	9A	EFFICIENCY SPIKES
2	9.80	4085.00	1.00	23.57	4108.57	30.866	9B	
3	19.16	2088.62	1.00	15.45	2104.07	31.053	9C	
4	49.19	813.25	1.00	10.90	824.17	32.376	9D	
5	98.26	407.09	1.00	9.18	416.27	34.147	9E	
6	195.38	204.75	1.00	8.42	213.17	37.285	9F	
7	438.71	91.18	1.00	7.90	99.08	44.553	9G	
(5 missing vials)								
13	311.98	128.22	1.00	8.17	136.39	40.940	1	EXPERIMENTAL SOLUTIONS
14	60.18	664.69	1.00	10.42	675.09	32.488	2	
15	68.07	587.76	1.00	10.17	597.93	33.066	3	
16	81.06	493.47	1.00	9.88	503.36	33.271	4	
17	48.75	820.59	1.00	11.57	832.16	32.429	5	
18	56.85	703.66	1.00	11.26	714.92	32.771	6	
19	59.68	670.27	1.00	10.59	680.88	32.748	7	
20	50.78	787.77	1.00	11.15	798.90	32.455	8	
21	49.01	816.20	1.00	10.71	826.91	32.348	9	
22	49.44	809.12	1.00	11.29	820.43	32.235	10	
23	49.73	804.56	1.00	11.26	815.83	32.226	11	
24	50.58	790.89	1.00	11.43	802.31	32.280	12	
25	4.81	8324.95	1.00	48.65	8373.60	31.140	0.05	REFERENCES
26	4.85	8260.62	1.00	36.70	8297.32	30.732	0.1	
27	4.78	8371.76	1.00	46.44	8418.20	31.080	0.2	
28	4.76	8404.41	1.00	43.28	8447.69	30.752	0.3	
29	4.79	8360.13	1.00	44.68	8404.80	30.857	0.4	
30	4.82	8300.62	1.00	44.61	8345.23	31.060	0.5	
31	4.76	8405.25	1.00	46.22	8451.47	30.922	0.6	
32	4.69	8543.28	1.00	49.89	8593.18	31.170	0.7	
33	4.78	8383.26	1.00	47.28	8430.54	30.693	0.8	
34	4.77	8398.74	1.00	43.19	8441.93	30.676	0.9	
35	4.80	8350.00	1.00	43.13	8393.13	30.686	1.0	
(1 missing vial)								
37	51.66	774.29	1.00	10.63	784.92	32.057	13	EXP. SOLUTIONS
38	49.53	807.59	1.00	11.71	819.32	32.341	14	
39	52.28	765.15	1.00	10.64	775.80	32.657	15	
40	51.20	781.41	1.00	10.80	792.19	32.234	16	
41	49.88	802.02	1.00	11.55	813.59	32.637	17	
42	48.32	827.88	1.00	11.53	839.42	32.567	18	
43	47.46	842.88	1.00	11.61	854.49	32.711	19	
44	999.98	16.98	1.54	2.32	19.29	54.025	BLANK	
(1 missing vial)								
46	999.98	16.97	1.54	2.25	19.22	53.724	BLANK	

```
SYSTEM NORMALIZED
C14 IPA DATA PROCESSED
C14 CHI SQUARE IPA DATA PROCESSED
H3 IPA DATA PROCESSED
H3 CHI SQUARE IPA DATA PROCESSED
BKG IPA DATA PROCESSED
```


4 Feb 93
PB

Data analysis of NaSrCl 0.5N: LSA data.

Plotting isotherm points depends upon:

1. Initial and final concentrations of Sr and Na in solution
2. wt. of zeolite used
3. CEC of zeolite
4. Volume of solution
5. conversion factors, i.e. wt Sr or Na / mole, ml to liters, etc

Only ① is affected by ISE, AA or LSA analyses. ②-⑤ remain constant throughout experiment and after initial measurement (weight of zeolite for instance) are not impacted by dilutions and $[Sr^{++}]$ or $[Na^{+}]$ measurement. Only ① is measured after exp. has begun all others are measured / determined prior to exp. run.

LSA impacts isotherm points generated from $[Sr^{++}]$ only

Sr data required:

- initial Sr concentration in solution
- final Sr concentration in solution

Some Assumptions:

1. Sr^{90} is added to all reference (initial Sr) solutions in equivalent amounts
2. Sr^{90} is sorbed in a similar fashion and at the same ratio as non-radioactive Sr, and Sr^{90} achieves chemical equilibrium with non-radioactive Sr during the experiment
3. Addition of Sr^{90} spike does not impact the overall Sr concentration (see p. 286 of this note book)
4. The ratio of the Sr^{90} change in solution is equivalent to the ratio of the non-radioactive Sr change:

$$\frac{Sr\text{-}90 \text{ initial}}{Sr\text{-}90 \text{ final}} = \frac{[Sr] \text{ initial}}{[Sr] \text{ final}}$$

4 Feb 93
PP

5. $SrCl_2 \cdot 6H_2O$ used to make reference solutions does not contain significant quantities of Sr-90. This can be verified by counting non-spiked Sr reference solutions. (see p. 300).

6. The final $[Sr^{++}]$ can be found by using:

$$Sr_{final} = \frac{Sr\ initial \cdot Sr\text{-}90\ final}{Sr\text{-}90\ initial}$$

where

Sr initial = calculated ppm Sr of references
Sr-90 initial = measured dpm of spiked reference solutions
Sr-90 final = measured dpm of exp. solutions

7. Initial Sr concentration (ppm) can be determined by:

- calculation from wt. $SrCl_2 \cdot 6H_2O$ added to ref. solutions

or - direct measurement using AAS

* because of the limited linear range of Sr analysis using AAS and the high concentrations of the reference solutions, AA dilution and measurement error can be substantial. Thus the initial ppm is determined by the wt. $SrCl_2 \cdot 6H_2O$ added. These concentrations were verified w/in 4% by AAS.

Initial concentration of exp. solutions is therefore the concentration of the reference solution added.

ex: ESr 0.05 used to make exp #1 solution

$\Rightarrow$ exp #1 Sr_i = 0.05 [Sr]

This entry is continued in notebook GC-09 CNWRA 060 p. 008

No further entries in this notebook. PP